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The Reduction of C* Bonds Proceeds with Retention of Configuration: Stereochemical Investigation of the Heterogeneous Reduction by Dideuterium of (Homohypostrophene)neopentyl(2-norbornyl)platinum(II) Complexes on Platinum Black.[1]

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Abstract: This paper reports an investigation of the heterogeneous, platinum(0)-catalyzed reductions by dideuterium of (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1** and (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**. The stereochemistries of bonding of the norbornyl groups to platinum are rigorously defined by crystal structures of **1** and **2**. The reductions occur on the surface of the catalyst: the organic ligands are converted to alkanes via reaction of surface alkyls with surface deuterides (D*); the platinum atom in the organometallic complex is reduced to platinum(0), and becomes part of the surface of the catalyst. Reduction of **1** with D_2 incorporates deuterium into the *exo*-2 position of norbornane; analogous reduction of **2** incorporates deuterium predominantly into the *endo*-2 position of norbornane. These results provide the most direct evidence now available that the stereochemistry of the reduction of C* bonds by H* (D*) proceeds

with retention of configuration. Approximately 20% of the *exo*-2-norbornyl* moieties undergo β-H activation at rates competitive with reductive elimination as norbornanes-d_n; in contrast, approximately 35% of the *endo*-2-norbornyl* moieties undergo α-H activation and epimerization to *endo*-2-norbornyl* at rates competitive with reductive elimination as norbornanes-d_n. These results are rationalized on the basis of steric interactions between the norbornyl moieties and the surface of platinum. The reduction by D_2 of homohypostrophene incorporates deuterium exclusively into the *exo* positions of the product tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane (HOPH); analogous reduction of 1 and 2 incorporates deuterium predominantly into the *endo* positions of HOPH. These results argue that the reduction of (diolefin)dialkylplatinum(II) complexes proceeds via adsorption of the platinum atom to the surface of the catalyst. Neopentane-d_1 is the major isotopomer of neopentane produced from the reductions of 1 and 2 by D_2.

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Introduction

The stereochemical outcome of heterogeneous hydrogenations of olefins on noble metal catalysts has been examined extensively.[2] Despite these efforts and the continuing progress in understanding the structures of hydrocarbons on metal surfaces,[3-24] the stereochemistry of reduction of the C* bond has been defined only by inference.[25] Determining the stereochemistry of reduction of C* bonds using the reduction of olefins requires a critical assumption since the *initial* stereochemistry of the C* bond is not known. Studies of the hydrogenation of olefins (that have no particular face selectivity) have showed that, over most metals, H_2 adds predominantly *cis* to the double bonds.[26-32] In addition, "anchoring" at sites remote from the double bonds increases the selectivity toward *cis* addition of H_2.[33-41] Several studies showed that *cis* addition of H_2 occurs to the less hindered face of the olefin.[42-49] These last reports constitute the most definitive, albeit indirect, characterization of the stereochemistry of reduction of C* bonds: the olefins *probably* coordinate by presenting their least hindered face to the surface of the metal; since H_2 adds to this face, the stereochemistry of the reduction of the C* bond proceeds with retention of configuration. We wanted to provide an independent and more direct determination of the stereochemistry of this reaction.

We have been studying the mechanisms of the heterogeneous hydrogenation of olefins and of organoplatinum compounds.[50-55] In this research, we showed that the heterogeneous, platinum-catalyzed hydrogenation of (diolefin)dialkyl-platinum(II) complexes (DOPtR$_2$) on platinum black produces diolefin-H_4, two equivalents of R-H, and platinum(0). The platinum(0) becomes part of the surface of the catalyst (eq 1). This reaction involves (i) adsorption of dihydrogen and the components of DOPtR$_2$ on the surface of the catalyst, (ii) generation of platinum-

$$\text{(diolefin-Pt-}R_2\text{)} \xrightarrow[\text{Pt (0)}]{3\,H_2} \text{(diolefin)} + Pt + 2\,RH \qquad (1)$$
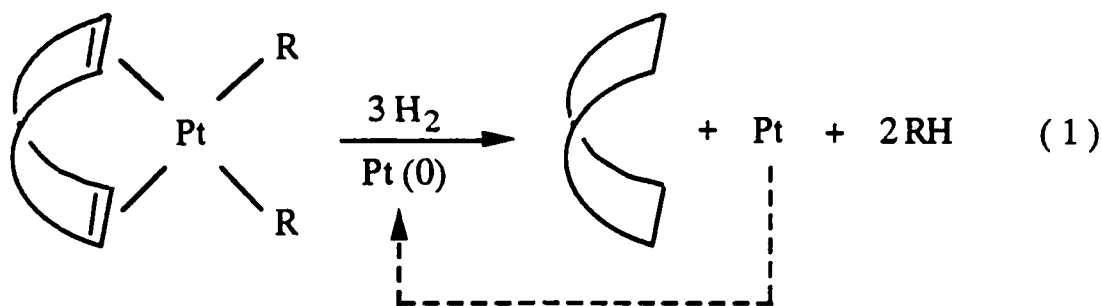
surface alkyls (DO∗ and R∗) from the alkyl and diolefin moieties originally present in the organometallic complex, and (iii) final reaction of the surface alkyls with surface hydrides to produce alkanes by reductive elimination. This reaction can be used to generate R∗ of known *initial* structure.[51,52] The intermediate surface alkyls generated from these reductions are related to those generated in heterogeneous hydrogenations of olefins (Scheme I).

In alkane solvents at relatively low temperatures (*ca* -20 °C) and high pressures of H_2 (*ca* 2.5 atm), the rate-determining step in production of alkane is an unspecified reaction occurring on the surface.[50] Under these conditions, the reduction by dideuterium of (1,5-cyclooctadiene)di-*n*-propylplatinum(II) produces 1-propane-d_1 as the major product, and that of (1,5-cyclooctadiene)di-*iso*-propylplatinum(II) produces 2-propane-d_1 as the major product. These observations suggest that the regiochemistries of the propyl-Pt moieties are preserved on transfer to the surface and on reduction.[52]

In the work presented here, we used this system to characterize the stereochemistry of the reduction of C∗ bonds in heterogeneous hydrogenations. We synthesized (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1** and (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**, and confirmed their structures using X-ray crystallography. We selected

Scheme I. Proposed Analogy Between Surface Alkyls Derived from Norbornene (left) and Those Derived from (Homohypostophene)neopentyl(*exo*-2-norbornyl)platinum(II) (right). HOP = Homohypostrophene; Np = Neopentyl.

HOPPtNp

H   H

H HOPPtNp H

H Np   HOP

H*   H*

+ Np-H + HOP-H$_4$
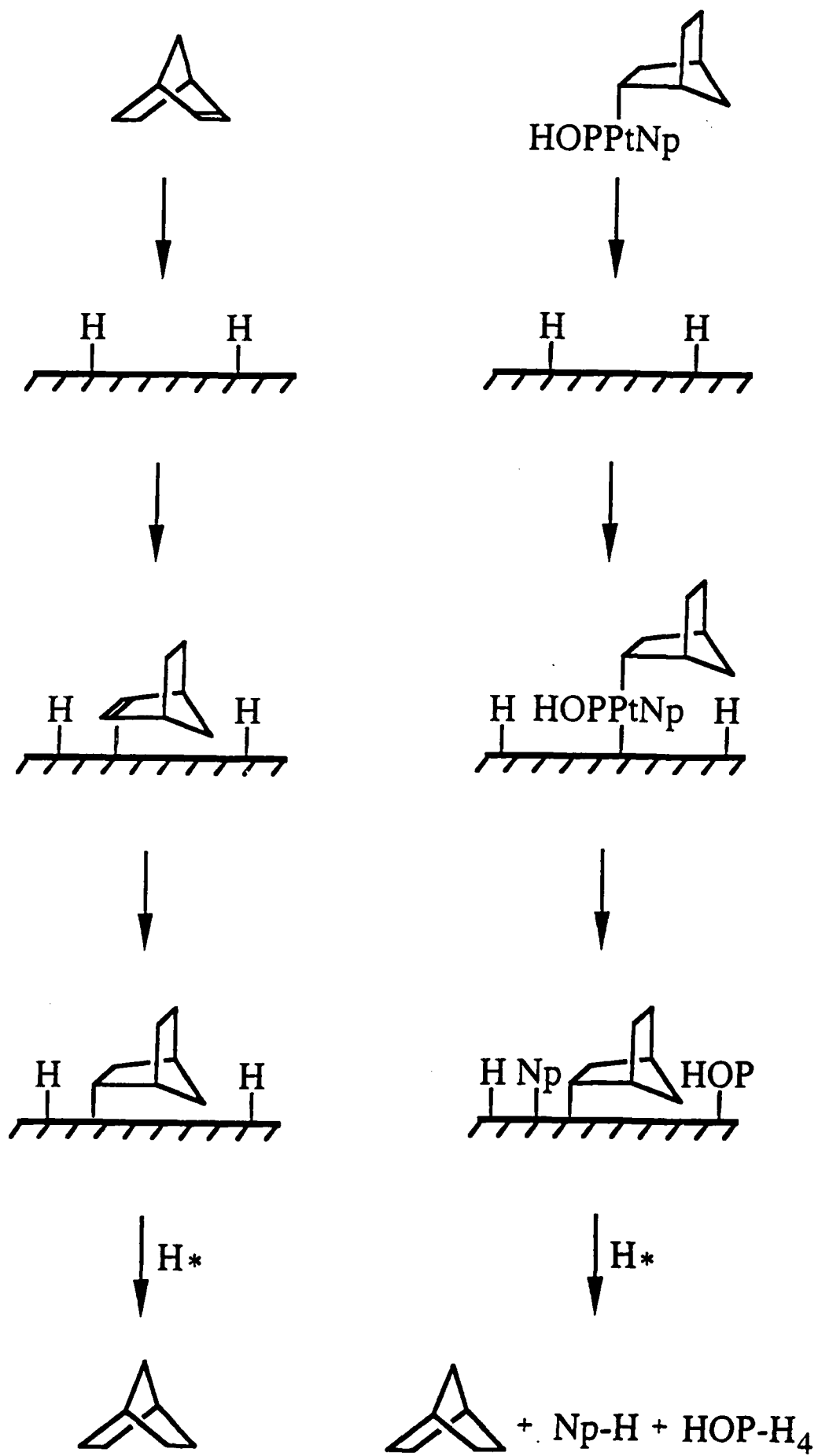
homohypostrophene (HOP) as the diolefin for three reasons. First, we could examine the stereochemistry of the reduction of the HOP moiety in HOPPtR$_2$. Second, HOP cannot form surface π-allyl groups, and is thus a relatively inert surface species that should not interfere in the reactions of coadsorbed alkyls.[56] Third, we could readily obtain crystals of complexes containing HOP. We chose 2-norbornyl groups as ligands for two reasons. First, norbornyl groups substituted at C(2) exist as two epimers (*exo* and *endo*, each enantiomeric); we wanted to synthesize both epimers to simplify interpretation of the data from reductions of the platinum complexes.[57] Second, the location of the deuterium atoms in the product norbornanes (*exo* vs *endo*) could be easily resolved using ^{1}H (or ^{2}H) NMR spectroscopy.[47,51,61] Neopentyl groups were chosen as ligands (rather than methyl groups) for two reasons. First, the reaction of mixtures of *exo-* and *endo-*2-norbornylmagnesium bromide (*ca* 45% *exo* and 55% *endo*) with HOPPtNpCl was selective ($\geq$ 90% 1 produced), but analogous reaction with HOPPtMeCl was not. Second, the substitution of neopentyl groups for methyl groups improved the crystallinity of DOPtR$_2$ complexes.

Reduction of these complexes with dideuterium over platinum black in alkane solvents generated deuterated norbornanes via intermediate 2-norbornyl* moieties of known stereochemistry. We determined the location of the deuterium atoms in these norbornanes using ^{1}H and ^{2}H NMR spectroscopy, and analyzed the alkanes produced in these reductions by GC/MS. Finally, ^{1}H NMR spectroscopy was used to locate the deuterium atoms in the homohypostrophanes produced in the reductions by D$_2$ of samples containing 1 and 2, and of free homohypostrophene.

Experimental Section

General. We purchased n-pentane (99+%, anhydrous, sure-seal bottle) from Aldrich, and stored it under argon. n-Heptane (Aldrich, 99.9%, HPLC grade) was distilled from Na/K, and diethyl ether (Mallinckrodt) was distilled from Na/benzophenone. We purchased platinum black (lot numbers 10410HT and 03019KT), neopentyl chloride (99%), *exo*-2-bromonorbornane (98%), norbornene (99%), benzophenone (99%), pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]-undecane-8,11-dione (98%), *tert*-butyllithium (1.7M in n-pentane), LiAlH$_4$ (1M in diethyl ether), and 10% AgNO$_3$ on silica gel from Aldrich, and used them without further purification. Di-μ-chloro-dichlorobis(ethylene)di-platinum(II) (Zeise's dimer, Strem), and dideuterium (99.5 atom % D, Matheson) were used as received. Cyclopentadiene was distilled from dicyclopentadiene (Aldrich, 97%), and benzoquinone (Baker) was recrystallized from petroleum ether.

We collected the ^{2}H NMR spectra on a Bruker WM 300 spectrometer operating at 46.03 MHz with broadband ^{1}H decoupling, and referenced to C$_6$D$_6$ (δ 7.15 ppm). Melting points were obtained in capillaries sealed under vacuum. We used a Hewlett Packard 5992A GC/MS (70 eV electron impact ionization) to measure mass spectra, and collected these data using the software for Selected Ion Monitoring from Hewlett Packard. The UV absorbance spectra of samples containing predominantly **1** and predominantly **2** were obtained with a Perkin-Elmer 552 spectrophotometer, and are included as supplementary material. We measured the UV absorbances of aliquots from kinetics runs on a Gilford 240 single-beam spectrophotometer at 286 and 292 nm for reductions of samples containing predominantly **1** and **2**, respectively (*vide infra*). The methods used to collect the X-ray structures of **1** and **2** are included as supplementary material. The lowest energy conformation and ^{1}H NMR coupling constants of

homohypostrophane (HOPH) were calculated with Macromodel V2.0 using the MM2(85) parameter set.[62] Oneida Research Co performed the elemental analyses.

Procedure for Reductions. Each reduction was performed as follows. A 20-mL pressure-bottle reactor (purchased from Lab Glass, and silanized as described previously[50]) was charged with 30 mg of platinum black and a football-shaped (10 x 6 mm) magnetic stirring bar. The vessel was capped with a neoprene septum, purged with argon, and immersed to within ~ 1 cm of its metal crown cap in a large bath of water/ethylene glycol (1:1, v:v) thermostatted by a Neslab Cryocool at -20 ± 1 °C. Solvent (1 mL) was added, and dideuterium was admitted to the reactor through a syringe needle inserted into the septum. The vessel was purged for 15 s, then pressurized to 2.4 atm (as monitored by inserting a syringe needle equipped with a pressure gauge through the septum of the reactor; the pressure reported is probably accurate to ± 5%). Stirring was started and maintained at 1800 RPM (the number of revolutions per minute of the magnetic stir bar as measured by a calibrated strobe light). After 10 minutes, we stopped the stirrer, allowed the catalyst to settle to the bottom of the vessel, and removed the solvent through a cannula. A yellow solution of the platinum complex (25 mg of dissolved in 4 mL of *n*-pentane or *n*-heptane)[63] was cooled to -20 °C and admitted to the vessel via cannula. We started the stirrer, and allowed the reaction to proceed for 90 min; after this time, we observed that the solution was clear, and that it showed no UV absorbance.

For reductions in *n*-pentane, the hydrocarbon products (with the exception of neopentane) were separated from the solvent by preparative GC on a F&M 700 instrument. We used a 1/4 in X 6 ft UCW-98 column operated at 150 °C with a helium flow of *ca* 30 mL/min. Elution times were: *n*-pentane, 30-90 s;

norbornane, 2-3 min; homohypostrophane (the product of the reduction of homohypostrophene) 30-35 min.

Kinetics of Reductions. Previous studies of the kinetics of reduction of $DOPtR_2$ in alkane[50] and protic solvents[53] under the conditions used here showed that for a wide variety of diolefins and R groups (where R is alkyl) the kinetic features---- rates of reduction and zero-order dependence on the concentration of substrate----were similar. Based on these observations, and because we had only limited quantities of 1 and 2, we did not perform an explicit investigation of the kinetics of reduction of these compounds. Nevertheless, since some platinum complexes, for example (norbornadiene)dimethylplatinum(II), react autocatalytically with H_2,[50] we needed to establish whether an autocatalytic reaction was important in the reductions of 1 and 2.

In order to test for autocatalysis, we ran blank reductions of mixtures of 1 and 2 in n-heptane without any catalyst present. Immediately after addition of the dissolved platinum complexes, we removed an aliquot $(t = 0)$[64] from the reactor. Stirring was resumed; simultaneously, a stopwatch was started. Every 15 min, an aliquot was removed until 90 min had elapsed. We diluted the aliquots under air by a factor of 100 by transferring 50 μL (using a 50 μL disposable glass micropipet) to a 5-mL volumetric flask, and filling it to the mark with solvent. The diluted solution was transferred to a 3.0-mL quartz cuvette (10 x 10 x 30 mm) using a disposable glass pipet. The UV absorbances did not diminish for either substrate during the 90-min period; therefore, autocatalysis did not contribute to the reduction of these compounds under the conditions employed here.

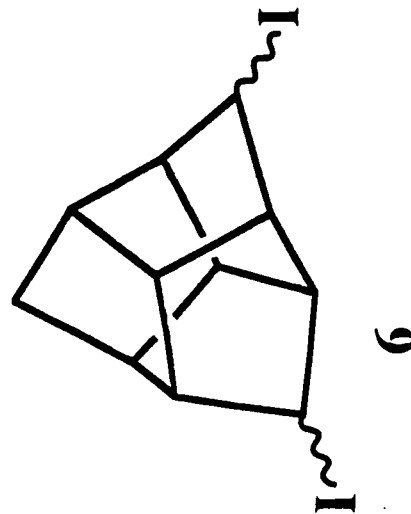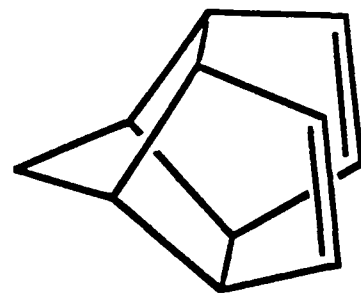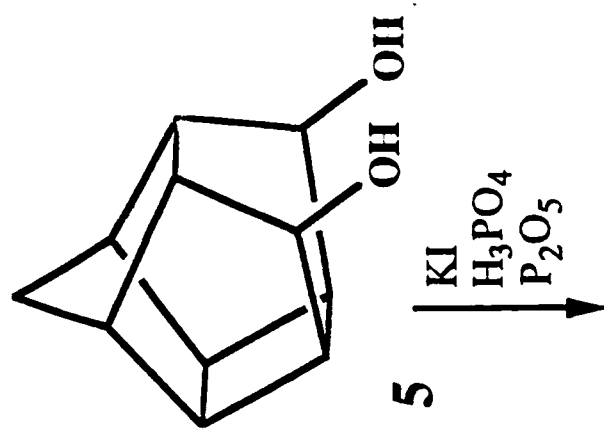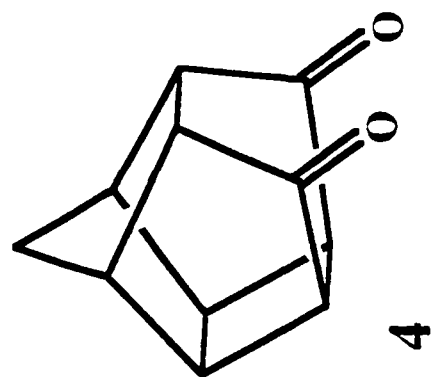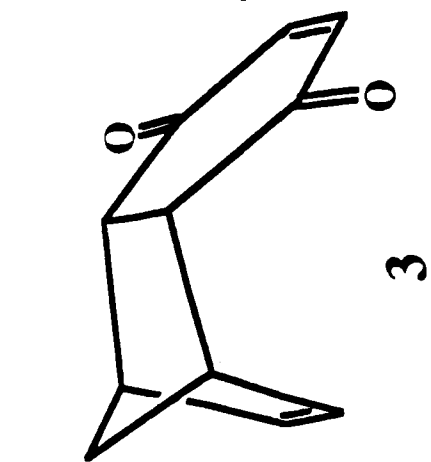**Synthesis of Tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undeca-2,6-diene (Homohypostrophene).** We synthesized homohypostrophene (*ca* 50-g scale) using the method of Smith and Barborak (Scheme II).[65] Since we observed that homohypostrophene decomposes to an insoluble white material on storage at -6 °C, we chose **6** as the immediate target of the large-scale synthesis, and converted this compound directly to homohypostrophene when desired.

1α, 4α, 4aβ, 8aβ-Tetrahydro-1,4-methanonaphthalene-5,8-dione, 3. From 154 mL of dicyclopentadiene (at 170 °C), we distilled 39 mL (470 mmol) of cyclopentadiene (at 44 °C) into a 50-mL graduated cylinder cooled to 0 °C. The cyclopentadiene was diluted into 90 mL of toluene at -78 °C. In a 1-L flask equipped with a magnetic stir bar, we dissolved 50.0 g (463 mmol) of benzoquinone in 370 mL of toluene. We added the cooled solution of cyclopentadiene dropwise to the stirred solution of benzoquinone via cannula, and periodically cooled the mixture with a dry ice/acetone bath. After the addition was complete, the solution was stirred for 34 h at room temperature. Removal of the solvent by rotary evaporation yielded 82 g of crude product whose ^{1}H NMR was consistent with that reported for **3**:[66] (CDCl$_3$, 250 MHz) δ 6.55 (s, 2 H), 6.05 (t, 2 H, J = 2 Hz), 3.53 (br s, 2 H), 3.20 (t, 2 H, J = 2 Hz), 1.47 (J_{AB} = 9 Hz, 2 H).

Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione, 4. We dissolved 82 g of **3** in a minimum amount of ethyl acetate, and irradiated this solution in a quartz tube for 8 h with a 450 watt Ace-Hanovia mercury arc lamp. Irradiation produced an off-white precipitate that was collected and recrystallized from acetone to give, after drying, 64.6 g of **4**. The ^{1}H NMR spectrum of the product was consistent with that of a sample of **4** purchased from Aldrich: (CDCl$_3$, 300 MHz) δ 3.17 (br s, 2 H), 2.92 (m, 2 H,), 2.80 (m, 2 H), 2.70 (s, 2 H), 1.95 (J_{AB} = 11 Hz, 2 H).

Scheme II. Synthesis of Homohypostrophene.

3     $\xrightarrow{h\nu}$ 4 $\xrightarrow{\text{LiAlH}_4}$ OH OH   5

$\xrightarrow[\substack{\text{H}_3\text{PO}_4 \\ \text{P}_2\text{O}_5}]{\text{KI}}$ I   I   6

$\xrightarrow{t\text{-BuLi}}$

Homohypostrophene

Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-diol, 5. Into a 2-L round-bottomed flask equipped with a magnetic stir bar, we transferred 64.6 g of the dione 4 using 500 mL of diethyl ether. To this stirred solution under argon, we added dropwise 800 mL of LiAlH$_4$ (1M solution in diethyl ether). The mixture was stirred overnight, then excess hydride was decomposed by the slow addition of 30 mL of H$_2$O followed by 60 mL of 15% NaOH solution. We added ca 200 mL of 50% H$_2$SO$_4$, and separated the phases. We extracted the aqueous phase with CHCl$_3$ (6 x 100 mL), combined the organic phases, and dried them over magnesium sulphate. Filtration followed by rotary evaporation yielded 63 g of crude 5. The ^{1}H NMR spectrum was consistent with that reported in the literature:[67] (CDCl$_3$, 250 MHz) δ 5.73 (s, 2 H; this resonance disappears upon addition of D$_2$O), 3.96 (br s, 2 H), 2.2-2.8 (m, 8 H) 1.37 (J_{AB} = 12 Hz, 2 H).

Diiodopentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane, 6. To a 2-L three-necked flask, we added 192 g of P$_2$O$_5$ and 253.5 g of 85% H$_3$PO$_4$. After the evolution of heat subsided, we added 364 g of KI, and 63 g of the diol 5. After attaching a mechanical stirrer, a reflux condenser, and a drying tube to the flask, we placed it in an oil bath at 108 °C for 12 h with stirring. The mixture was allowed to sit at room temperature for ca 12 h, after which it was transferred to a 2-L separatory funnel using diethyl ether and H$_2$O. The layers were separated, and the aq phase was extracted with diethyl ether (2 x 500 mL). We washed the combined organic phases with 10% aq Na$_2$S$_2$O$_3$, and twice with H$_2$O. After drying the organic phase with magnesium sulphate, we added decolorizing carbon (Norit), and filtered the solution. Removal of the solvent by rotary evaporation provided a yellow oil. Trituration of this oil with acetone gave, after drying under vacuum, 89.3 g (224 mmol, 48% yield based on benzoquinone) of a white solid; the ^{1}H NMR spectrum of this material was consistent with that reported for a mixture of

stereoisomers, *syn*-4, *anti*-7- and *syn*-4, *syn*-7-diiodopentacyclo-[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane:[65] (CDCl$_3$, 300 MHz) δ 3.95 (br s, 2 H), 2.0-3.4 (complex m, 8 H), 1.45 (m, 2 H).

Homohypostrophene. Under an atmosphere of argon, a flame-dried 100-mL round-bottomed flask containing a magnetic stir bar was charged with 5.03 g (12.6 mmol) of **6** and 50 mL of diethyl ether. To this solution, we slowly added 15 mL (26 mmol) of *tert*-butyllithium (1.7*M* in *n*-pentane) via cannula. The solution was stirred for 10 min after the addition was completed. We added H$_2$O carefully to quench any excess lithium reagent. A sufficient amount of water was then added to produce a clear biphasic solution. We extracted the aqueous phase with diethyl ether, combined the organic extracts, and dried them with magnesium sulphate. Filtration followed by careful rotary evaporation at 0 °C yielded a waxy white semi-solid which was sublimed. The sublimate was chromatographed on 10% AgNO$_3$ on silica gel. Elution with *n*-pentane provided 0.247 g of pentacyclo-[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (trishomocubane).[65] ^{1}H NMR: (CDCl$_3$, 300 MHz) δ 1.98 (m, 8H), 1.30 (s, 6H). Elution with 1:1 *n*-pentane/diethyl ether produced, after careful removal of the solvent, 0.885 g (6.1 mmol, 48% yield) of homohypostrophene.[65] ^{1}H NMR (CDCl$_3$, 250 MHz): δ 5.90 (s, 2 H), 3.14 (s, 2 H), 2.32 (s, 4 H), 1.63 (s, 2 H).

Tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane, (Homohypostrophane, HOPH). Homohypostrophane (HOPH) is the product from the reduction of homohypostrophene with H$_2$ over platinum black. Following the procedure for reductions outlined above, we hydrogenated 0.104 g (0.72 mmol) of homohypostrophene in *n*-pentane. The solution was separated from the catalyst using a pipet, and the solvent was carefully removed by a flow of argon.

Sublimation of the resulting white solid gave 0.077 g (0.52 mmol; 72%) of HOPH as a waxy white solid. Mp: sub. MS: m/e (rel. int.) 148 (38) M+, 119 (49), 91 (55), 81 (51), 80 (64), 79 (76), 77 (40), 67 (100), 66 (95), 41 (76), 39 (94), 27 (68). ^{1}H NMR (CDCl$_3$, 500 MHz, Figure 7):[68] δ 2.06 (m, 2 H, H$_a$), 1.98 (br s, 4 H, H$_b$), 1.56 (br d, 4 H, J = 8 Hz, H$_c$), 1.46 (s, 2 H, H$_d$), 1.45 (br d, 4 H, J ~ 8 Hz, H$_e$). ^{13}C NMR (CDCl$_3$, 100.6 MHz): 48.1, 43.0, 30.4, 25.5. Anal. Calcd. for C$_{11}$H$_{16}$: C, 89.12; H, 10.88; Found: C, 88.85; H, 10.90.

Synthesis of Grignard Reagents. Neopentylmagnesium chloride. Into a 500-mL round-bottomed flask containing a magnetic stir bar, we placed 10.0 g (0.411 mol) of magnesium chips. We wired a rubber septum onto the flask, and flame-dried the flask under a flow of argon. After the flask cooled, we added *ca* 300 mL of diethyl ether, initiated stirring, and slowly added via cannula 30.0 mL (0.244 mol) of neopentyl chloride in *ca* 50 mL diethyl ether. We replaced the septum with a reflux condenser, and refluxed the solution for *ca* one week under argon. Titration[69] showed that the solution was 0.6M in neopentylmagnesium chloride (*ca* 74% yield).

2-Norbornylmagnesium bromide. We synthesized this Grignard reagent several times using the following procedure. We placed 5.0 g (0.206 mol) of magnesium chips and a magnetic stir bar to a 200-mL round-bottomed flask. The flask was capped with a rubber septum, and flame-dried under argon. Diethyl ether (*ca* 80 mL) was added, stirring was started, and 10.0 mL (0.078 mol) of *exo*-2-bromonorbornane in *ca* 20 mL diethyl ether was added via cannula. A slow rate of addition was maintained so that the temperature of the flask was warm, but the solution was not refluxing. After the addition was complete, the solution was stirred for one hour, then allowed to sit overnight.

Titration[69] of these solutions typically showed the solutions to be 0.6-0.7M in 2-norbornylmagnesium bromide (ca 60-70% yield). According to prior reports, these solutions contained a mixture of ca 40% exo- and 60% endo-2-norbornyl-magnesium bromide.[58,60]

endo-2-Norbornylmagnesium bromide. This Grignard reagent was synthesized using a variation on established procedures.[58,60] We transferred under argon 50.0 mL (30.0 mmol) of a 0.6M solution of 2-norbornylmagnesium bromide to a flame-dried, 100-mL Schlenk flask capped with a rubber septum, and containing a magnetic stir bar. The flask was placed in a -10 °C salt/ice-water bath, and stirring was initiated. We added via cannula a solution of 3.28 g of benzophenone (18.0 mmol, 0.6 equiv) in 10 mL of diethyl ether. The solution rapidly turned dark pink; a white precipitate was observed. The solution was allowed to stir for 5 min at -10 °C, after which the flask was placed in a dry ice/acetone bath at -78 °C. We attached a medium glass frit having male ground-glass joints at both ends to a 100-mL Schlenk flask. This filtration apparatus was flame-dried under a purge of argon. After cooling, we attached the apparatus under a flow of argon to the Schlenk flask containing the dark-pink solution. The solution was filtered quickly through the frit; the filtrate, presumably containing endo-2-norbornylmagnesium bromide,[58,60] was stored at -78 °C, and used shortly thereafter.

Synthesis of Platinum Complexes. (Homohypostrophene)-platinum(II)dichloride, HOPPtCl$_2$. Under argon, we added 3.51 g of a 3:1 mixture of homohypostrophene/trishomocubane (ca 18 mmol of homohypostrophene) in 25 mL of benzene to an orange suspension of 5.0 g (8.5 mmol) of Zeise's dimer in 100 mL of benzene in a 250-mL round-bottomed flask

equipped with a magnetic stir bar. Stirring for 24 h at room temperature produced a precipitate of white needles in a dark brown solution. We collected the precipitate by filtration through a medium glass frit, and washed it with benzene. Recrystallization of the precipitate from hot chloroform yielded 3.68 g (8.97 mmol) of HOPPtCl$_2$ as white needles (53% yield based on platinum). Mp: 252-300 °C dec. ^{1}H NMR (CDCl$_3$, 400 MHz): δ 6.38 ("t" with Pt satellites, J_{Pt-H} = 78 Hz, 4H), 3.27 ("t" with Pt satellites, J_{Pt-H} = 26 Hz, 4H) 3.21 (m, 2 H), 1.85 (s, 2 H). Anal. Calcd for C$_{11}$H$_{12}$PtCl$_2$: C, 32.21; H, 2.95. Found: C, 32.22; H, 2.65.

(Homohypostrophene)platinum(II)diiodide, HOPPtI$_2$. The diiodide was obtained in quantitative yield from HOPPtCl$_2$ by suspending the dichloride in acetone, saturating the solution with KI (the solution turned from clear to yellow immediately upon addition of KI), and stirring for three days. Aqueous workup and extraction with chloroform followed by recrystallization from hot chloroform yielded HOPPtI$_2$ as yellow needles. Mp: 247-285 °C dec. ^{1}H NMR (CDCl$_3$, 500 MHz): δ 6.47 ("t" with Pt satellites, J_{Pt-H} = 78 Hz, 4H), 3.30 (sep, J = 1.8 Hz, 2 H), 3.15 ("t" with Pt satellites, J_{Pt-H} = 27 Hz, 4H), 1.93 (s, 2 H). Anal. Calcd for C$_{11}$H$_{12}$PtI$_2$: C, 22.28; H, 2.04. Found: C, 22.25; H, 2.02.

(Homohypostrophene)dineopentylplatinum(II), HOPPtNp$_2$. This compound could be synthesized from either the corresponding dichloride or diiodide; we present here a representative example. In a flame-dried 100-mL Schlenk flask equipped with a magnetic stir bar, a suspension of 0.902 g (1.88 mmol) of HOPPtI$_2$ in diethyl ether (50 mL) was cooled to -78 °C under an atmosphere of argon. We added a 0.6M solution of neopentylmagnesium chloride (7.2 mL, 4.3 mmol) dropwise via cannula. The solution was stirred and allowed to warm slowly to 0 °C. Analysis by TLC (1:1 n-pentane/diethyl ether) showed the

reaction to be complete. We added excess H_2O slowly to quench excess Grignard reagent. The aqueous phase was extracted with diethyl ether, and the extracts dried over magnesium sulfate. Decolorizing carbon (Norit) was added, and the solution was filtered into a 250-mL round-bottomed flask. We concentrated the solution to dryness on a rotary evaporator, and obtained a yellow-green solid. This solid was chromatographed on silica gel using n-pentane as the eluant. The first fraction off the column (yellow, absorbs in the UV) contained the product. Removal of solvent followed by recrystallization from diethyl ether/methanol yielded yielded 0.683 g (1.42 mmol; 76% yield) of $HOPPtNp_2$. Mp: 106-107 °C. ^1H NMR (C_6D_6, 400 MHz): δ 5.62 ("t" with Pt satellites, J_{Pt-H} = 51 Hz, 4H), 2.66 (sep, J = 1.8 Hz, 2 H), 2.43 (br s, 4 H), 2.12 ("t" with Pt satellites, J_{Pt-H} = 92 Hz, 4H), 1.37 (s, J_{Pt-C} = 124 Hz, 18 H), 1.09 (s, 2 H). Anal. Calcd for $C_{21}H_{34}Pt$: C, 52.37; H, 7.12. Found: C, 52.63; H, 6.95.

(Homohypostrophene)neopentylplatinum(II)chloride, HOPPt(Np)Cl. In a 50-mL round-bottomed flask equipped with a magnetic stir bar, we dissolved 1.05 g (2.18 mmol) of $HOPPtNp_2$ in a minimum amount of n-pentane, and added 1 mL of conc HCl. After stirring for 2 h at room temperature, a white precipitate had formed, and analysis by TLC (1:1 n-pentane/diethyl ether) indicated that the reaction was complete. The solution was neutralized by adding saturated sodium bicarbonate solution. We extracted the aqueous phase with diethyl ether and dried the organic phase over magnesium sulfate. After filtration, rotary evaporation yielded an off-white solid that was chromatographed on silica gel (1:1 n-pentane/diethyl ether), and recrystallized from diethyl ether to afford glassy pale yellow plates of HOPPt(Np)Cl (0.888 g, 1.99 mmol; 91% yield). Mp: 148-149 °C. ^1H NMR ($CDCl_3$, 500 MHz): δ 6.40 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 44 Hz, 2H), δ 5.20 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 91 Hz, 2H), 3.23 (m, 1 H), 3.10

(m, 1 H), 3.04 (br m, 2 H), 2.91 (br m, 2 H), 1.66 ("t" with Pt satellites, J_{Pt-H} = 76 Hz, 2 H), 1.59 (br s, 2 H), 1.07 (s, J_{Pt-C} = 124 Hz, 9H). Anal. Calcd for $C_{16}H_{23}PtCl$: C, 43.10; H, 5.20; Found: C, 43.21; H, 4.99.

(Homohypostrophene)neopentylplatinum(II)iodide, HOPPt(Np)I.
This compound was obtained from the corresponding chloride in quantitative yield by dissolving the chloride in a minimum amount of acetone, saturating the solution with KI (the solution turns from clear to yellow immediately upon addition of KI), and stirring for two days. Aqueous workup and extraction with diethyl ether followed by recrystallization from hot diethyl ether yielded HOPPt(Np)I as yellow prisms. Mp: 143-144 °C. ^{1}H NMR (CDCl$_3$, 500 MHz): δ 6.41 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 48 Hz, 2H), δ 5.35 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 89 Hz, 2H), 3.26 (m, 1 H), 3.10 (m, 1 H), 2.97 (br m, 2 H), 2.91 (br m, 2 H), 2.07 ("t" with Pt satellites, J_{Pt-H} = 81 Hz, 2 H), 1.63 (br s, 2 H), 1.09 (s, J_{Pt-C} = 124 Hz, 9H). Anal. Calcd for $C_{16}H_{23}PtI$: C, 35.76; H, 4.31; Found: C, 35.87; H, 4.21.

(Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.
Under argon, we added via cannula 30.0 mL (18.0 mmol) of a 0.6M solution of 2-norbornylmagnesium bromide (ca 45% exo and 55% endo) in diethyl ether to a flame-dried 100-mL Schlenk flask equipped with a magnetic stir bar. We placed the flask in a -10 °C salt/ice-water bath, and added dropwise via cannula a yellow solution containing 0.252 g (0.565 mmol) of HOPPt(Np)Cl in diethyl ether. After one h, the solution had darkened; analysis by TLC (1:1 diethyl ether/n-pentane) showed that the reaction was complete. We added H$_2$O to destroy the excess Grignard reagent, extracted the aqueous phase with diethyl ether, combined the organic phases, and dried them over magnesium sulphate. After adding decolorizing carbon (Norit), the solution was filtered into a round-bottomed flask,

and concentrated to a yellow oil. This oil was chromatographed on silica gel using *n*-pentane as the eluant. The first few fractions (yellow, absorbs in the UV) were collected, and evaporated to dryness. The resulting yellow solid, recrystallized from diethyl ether/methanol, gave 83 mg (0.164 mmol, 29% yield) of a mixture of **1** (96%) and **2** (4%).[70] Mp: 98-120 °C dec. ^{1}H NMR (C_6D_6, 500 MHz): δ 5.55-5.80 (complex m, 4 H), 2.6-2.7 (complex m, 2 H), 2.35-2.55 (complex m, 6H), 2.02 (m, 1 H), 1.88-1.98 (complex m, 1 H),1.88 & 1.85 (J_{AB} = 11 Hz, 2 H), 1.79 (m, 1 H), 1.63 (m, 1 H), 1.51 (m, 2 H), 1.40 (m, 1 H), 1.35 (s, 9 H), 1.28 (m, 1 H), 1.09 (s, 2 H). ^{13}C NMR (C_6D_6, 125.8 MHz): 108.0, 107.3, 106.9, 105.8, 69.23, 69.18, 54.6, 54.0, 53.9, 53.8, 53.5, 46.4, 43.3, 42.4, 41.5, 41.3, 39.7, 38.8, 37.5, 36.13, 36.06, 35.97, 29.9. Anal. Calcd. for $C_{23}H_{34}Pt$: C, 54.64; H, 6.78; Found: C, 54.62; H, 6.56.

A similar synthesis on somewhat larger scale (0.256 g, 0.574 mmol HOPPt(Np)Cl) produced 0.163 g (32.2 mmol, 58% yield) of a mixture of **1** (90%) and **2** (10%). We used this mixture for the isotopic reductions.

(Homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II), 2.
A flame-dried, septum-capped 200-mL round-bottomed flask equipped with a magnetic stir bar was charged with 0.253 g (0.471 mmol) of HOPPt(Np)I in a minimum amount of diethyl ether, and cooled under argon to -10 °C in a salt/ice water bath. To this stirred solution, we added via cannula the solution containing the *endo*-2-norbornylmagnesium bromide (*ca* 18 mmol; *vide supra*). Over the course of 1 hr at -10 °C, the solution turned from yellow to brown. Analysis by TLC (1:1 *n*-pentane/diethyl ether) showed the presence of starting material (R_f ~ 0.5), and possible product (R_f ~ 0.9). Elution with *n*-pentane showed that the spot at R_f ~ 0.9 had several components. We quenched the reaction by adding H_2O.

The aqueous phase was extracted with diethyl ether; the extracts were combined and dried with magnesium sulphate. We added decolorizing carbon (Norit), and filtered the solution into a round-bottomed flask. Rotary evaporation yielded a yellow oil which was chromatographed on silica gel using n-pentane as the eluant. The first yellow fractions showed absorbance in the UV, and contained the desired product. Recrystallization from diethyl ether/methanol yielded 30 mg (0.059 mmol, 13% yield) of 2 as yellow needles. Mp: 111-180 °C dec. ^{1}H NMR (C_6D_6, 500 MHz): δ 5.79 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 52 Hz, 2 H), 5.62 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 48 Hz, 1 H), 5.55 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 52 Hz, 1 H), 3.03 (br s, 1 H), 2.80 ("t" with Pt satellites, J_{Pt-H} = 96 Hz, 1H), 2.66 (m, 2 H), 2.47 (m, 2 H), 2.42 (m, 2 H), 2.37 (m, 1 H), 1.99 & 1.84 (J_{AB} = 11 Hz, 2 H), 1.92 (complex m, 3 H), 1.70 (m, 2 H), 1.35-1.55 (complex m, 4H), 1.33 (s, 9 H), 1.09 (s, 2 H). Anal. Calcd. for $C_{23}H_{34}Pt$: C, 54.64; H, 6.78; Found: C, 54.58; H, 6.60.

Two subsequent syntheses produced 0.091 g (0.18 mmol, 39% yield) of a mixture of 2 (98%) and 1 (2%), and 0.062 g (12 mmol, 26% yield) of a mixture of 2 (97%) and 1 (3%).[70] We used the last mixture for the isotopic reductions.

Isotopic Analysis of Alkanes-d_n. For analyses by GC/MS, we used the average content of deuterium, d_{av} (eq 2), to describe the isotopic compositions of

$$d_{av} = 1/100 \sum_{n=1}^{m} n \ (\% \ \text{alkane-}d_n) \qquad (2)$$

the alkanes produced in the reductions.[51-53] In analyses by NMR, the values of d_{av} simply reflect the content of deuterium derived from integrations (*vide infra*). We believe that all values of d_{av} are accurate to ± 5% absolute.

Isotopic analyses by GC/MS were conducted using procedures analogous to those described earlier.[51-53] The relevant mass spectral data (m/e (rel. int.)) are: for norbornane, 96 (100.0) M^+, 95 (29.7), 97 (7.2); for HOPH, 148 (100.0) M^+, 149 (8.7). Distributions of the ions from norbornane were corrected for $(M - 1)^+$ by iteratively subtracting from the $(n - 1)$th peak the $(M - 1)^+$ percentage of the corrected value for the (n)th peak, and normalizing the resulting distribution. Distributions of ions for both molecules were corrected for natural abundance ot ^{13}C by iteratively subtracting from the nth peak the $(M + 1)^+$ percentage of the corrected value for the $(n - 1)$th peak, and normalizing the resulting distribution.[71] No other fragment ions with relative abundances > 1.0 fell within the range of relevant m/z.

In analyses by ^1H NMR, standard integration techniques using Bruker software were used to determine the isotopic content of norbornane-d_n and HOPH-d_n. For analyses of norbornane-d_n, we integrated H_b and ($H_c + H_d$) relative to H_a (Fig 4); for analyses of HOPH-d_n, we integrated ($H_d + H_e$) and H_c relative to H_a and H_b (Fig 7). We used a 10 s relaxation delay (relaxation delay + acquisition time totalled 13 s) to acquire the spectra.

We used an acquisition time of 2 s to collect the ^2H spectra of the norbornanes. No relaxation delay was employed. The resonances at δ 1.12 and 1.42 were integrated relative to each other by enlarging the printed spectra, cutting out the peak areas, and weighing them. Triplicate analyses of each spectrum differed by no more than 0.7%.

Results and Discussion

X-Ray Crystal Structures. Figures 1 and 2 are ORTEP plots of the structures of **1** and **2**. These structures are detailed in the Supplementary Material to this paper. Each compound clearly possesses a unique stereochemistry of bonding of the norbornyl group to platinum. The fact that the thermal ellipses in Figure 1 are larger than those in Figure 2 might reflect the fact that we collected the structure of **1** at 0 °C, and the structure of **2** at -58 °C.

NMR Spectra of the Norbornanes. Figure 3 shows the ^{1}H and ^{2}H NMR spectra of the norbornanes resulting from the reductions by D_2 of samples containing 90% **1** and 10% **2**, and 97% **2** and 3% **1**. These data show that the reduction of **1** incorporates deuterium into the *exo* position of norbornane, and the reduction of **2** incorporates deuterium predominantly into the *endo* position of norbornane. Assuming that the stereochemistry of bonding of the norbornyl moieties to platinum is maintained upon transfer to the surface(*vide infra*), these results argue that the reduction of C∗ bonds proceeds with predominant retention of configuration.

Formation of 2-Norbornyl∗ Occurs Without Loss of the Stereochemistry of Bonding Between the 2-Norbornyl Moieties and Platinum(II). Stereochemical[51] and kinetic[50] data provide support for this contention: the stereochemistry of reduction of the diolefin moieties of DOPtR$_2$ complexes indicate that the mechanism for reduction of these complexes occurs by initial adsorption at platinum (*vide infra*). Adsorption at platinum should not invert the stereochemistry of the norbornyl-Pt bond.

Figure 1. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1**. The molecule crystallizes in a non-centrosymmetric space group; the absolute configuration (*R*) was determined crystallographically. The data for this structure were collected at 0 °C.

Figure 2. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**. The molecule crystallizes in a centrosymmetric space group; the *S* enantiomer is shown. The data for this structure were collected at -58 °C.

Figure 3. ^{1}H NMR spectra (C_6D_6, 500 MHz) of (i) norbornane-d_0, and the norbornanes from the reduction by D_2 of samples containing (ii) 97% **2** and 3% **1**, and (iii) 90% **1** and 10% **2**. ^{2}H NMR spectra (C_6D_6, 46.03 MHz) of the norbornanes from the reductions by D_2 of samples containing (iv) 97% **2** and 3% **1**, and (v) 90% **1** and 10% **2**.

(v)

(iv)

(iii)

(ii)

(i)

PPM

The rate determining step in the heterogeneous hydrogenations of DOPtR$_2$ complexes has not been unambiguously identified, but the activation energy for the reduction of (1,5-cyclooctadiene)dimethylplatinum(II) (CODPtMe$_2$) over platinum black in n-heptane is 15 ± 2 kcal/mol,[50] and that for inversion at a methyl carbon (e.g. S$_N$2 displacement on MeI and MeBr) is typically 15-20 kcal/mol.[72] The similar magnitude of these activation energies suggests that if inversion at carbon o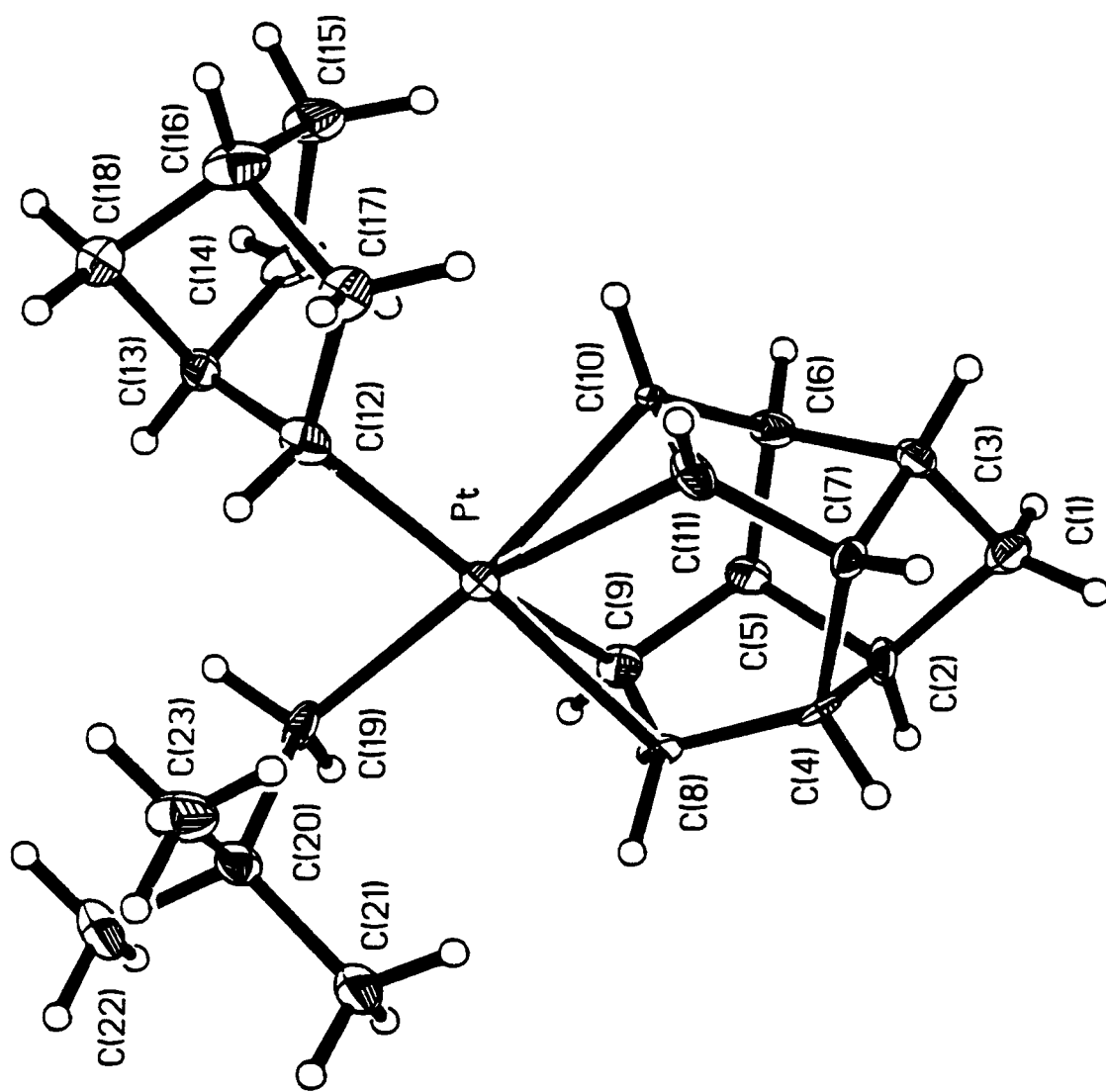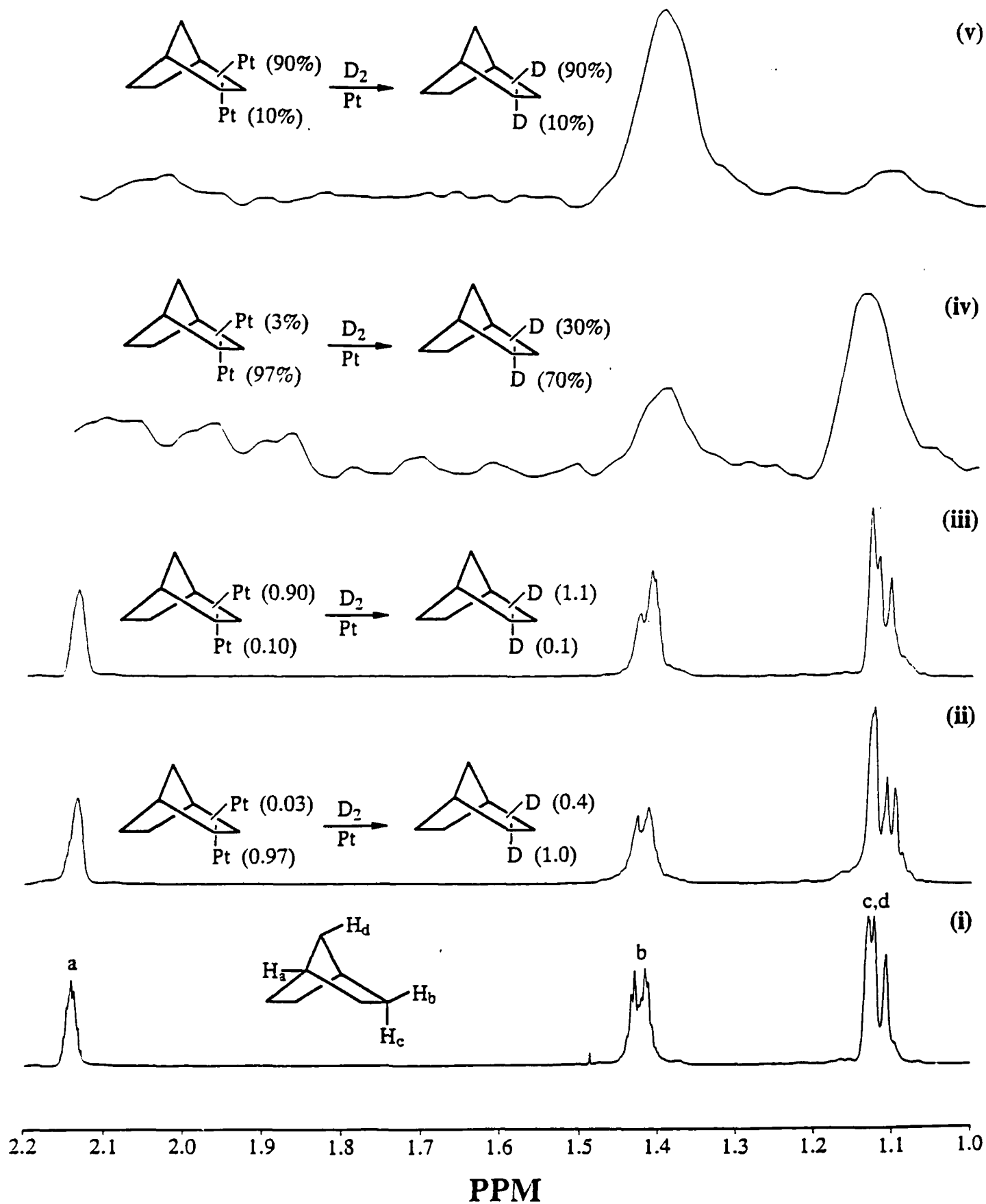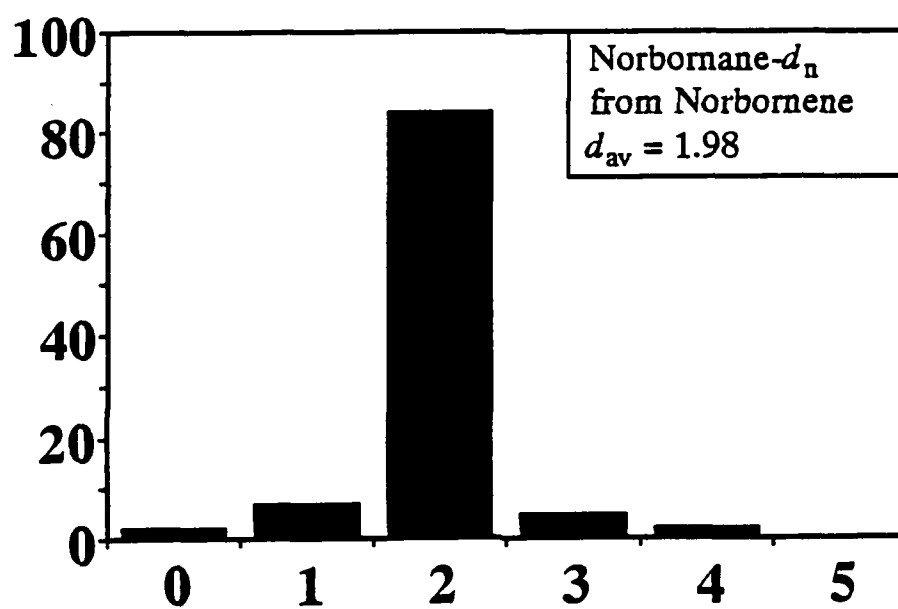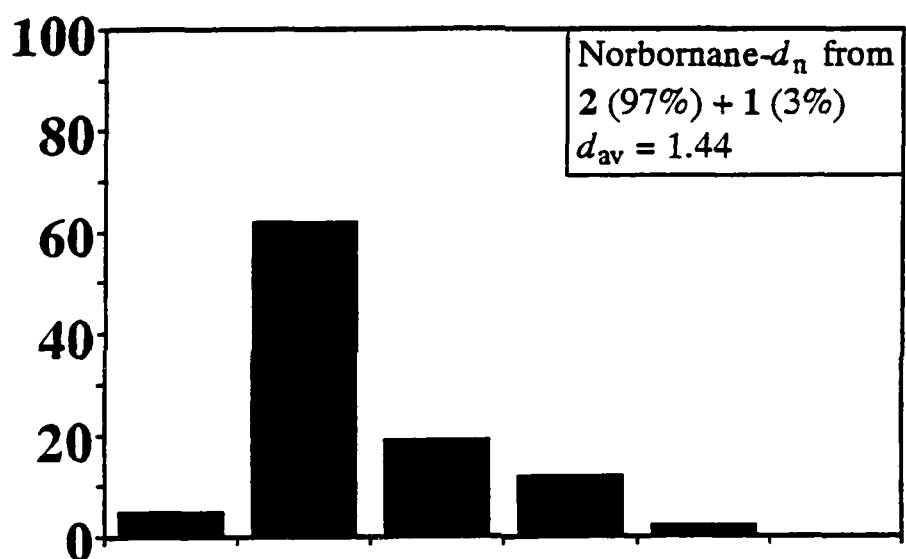ccurs in the reductions of DOPtR$_2$ complexes, the rates of these reductions should be influenced by structure in ways similar to those well established for S$_N$2 reactions. In fact, the reductions of CODPtR$_2$ complexes and S$_N$2 displacements on alkyl iodides (taken as a representative set) follow very different patterns of relative rates. For the former reaction, the relative rates of reduction are CODPtMe$_2$ (1.0), CODPtEt$_2$ (1.0), CODPt(iso-Pr)$_2$ (0.69), CODPt(iso-Bu)$_2$ (0.40), CODPtNp$_2$ (0.23), and CODPtPh$_2$ (0.60);[50] for the latter, the relative rates of displacement by Cl$^-$ with inversion at carbon are MeI (1.0), EtI (0.090), iso-PrI (0.0029), iso-BuI (0.0034), NpI (0.0000013), and PhI (0.0).[72] The absence of a correlation between the rates of reduction of DOPtR$_2$ complexes and the rates of inversion at carbon in S$_N$2 reactions is compatible with the hypothesis that the mechanism for the reduction of the platinum complexes involves retention at C1 of the R group in the reaction R-Pt ----> R*.

Mass Spectral Analysis of the Norbornanes. Figure 4 provides the mass spectral data for the norbornanes produced in the reductions by D$_2$ of norbornene, and samples containing 90% 1 and 10% 2, and 97% 2 and 3% 1. Norbornane-d_1 is the major product from reductions of the platinum complexes, and norbornane-d_2 is the major product from the reduction of norbornene.

Figure 4. Isotopic distributions determined from the mass spectra of the norbornanes produced from the reductions by D_2 of norbornene (bottom), a mixture of 97% **2** and 3% **1** (middle), and a mixture of 90% **1** and 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Norbornane-d_n from
1 (90%) + **2** (10%)
$d_{av} = 1.25$

Norbornane-d_n from
2 (97%) + **1** (3%)
$d_{av} = 1.44$

Norbornane-d_n
from Norbornene
$d_{av} = 1.98$

Percent Composition of Isotopomers
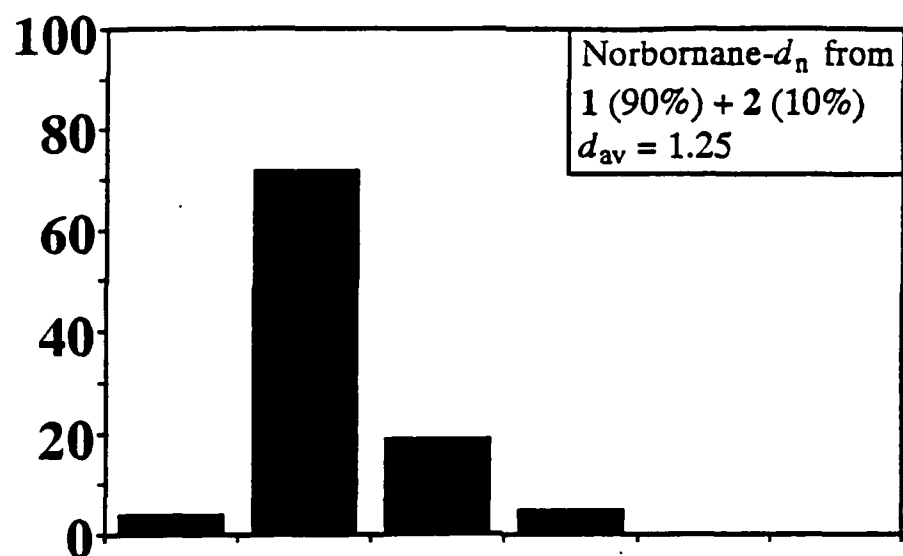
Number of Deuteriums

Table I gives the values of d_{av} determined from the mass spectral and ^{1}H NMR analyses of these norbornanes, and shows that the results from both analytical methods are in good agreement. The fact that these numbers agree indicates that activation of H_a and H_d (more correctly, incorporation of deuterium into these positions) does not occur. The norbornanes produced from both platinum complexes contain more than 1.0 D (the number of equivalents of deuterium expected based on the stoichiometry of the reaction). The incorporation of excess deuterium probably results from either (or both) α-H activation, or β-H activation (β-H elimination) prior to reductive elimination of 2-norbornyl* moieties from the surface (*vide infra*). Since it is unlikely under these conditions that every activation of a C-H bond results in subsequent incorporation of deuterium, we define an activation-incorporation event as an incorporation (e.g. α incorporation or β incorporation).

The isotopic composition of the norbornanes produced from the reductions by D_2 of samples containing 1 and 2 are summarized in eq 3 and 4. Equation 3

(3)

(4)
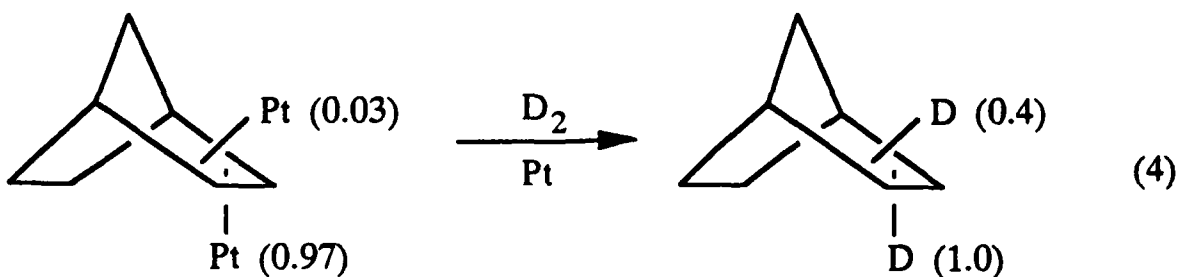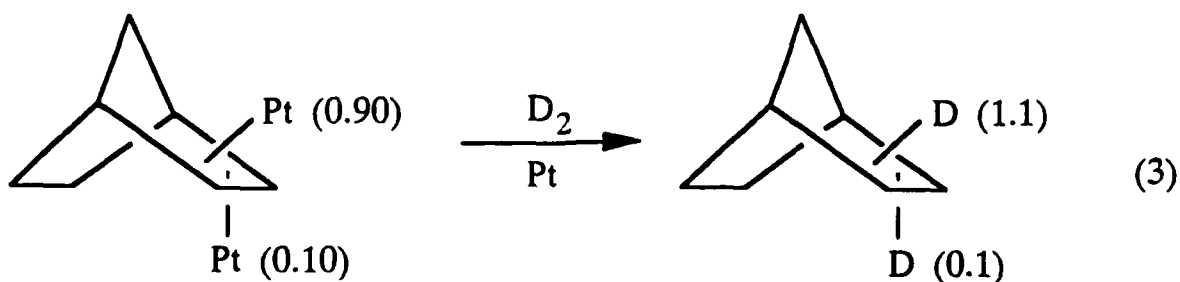
Table I. Isotopic Compositions (d_{av}) of the Alkanes-d_n from the Reductions by D_2 of **1, 2**, Homohypostrophene, and Norbornene.[a]

Alkane-d_n	Substrate	d_{av} (MS)	d_{av} (^{1}H NMR)
Homohypostrophane	**1** (90%) + **2** (10%)	5.22	5.26
	2 (97%) + **1** (3%)	5.24	5.30
	homohypostrophene	3.88	3.84
Norbornane	**1** (90%) + **2** (10%)	1.25	1.17
	2 (97%) + **1** (3%)	1.44	1.35
	norbornene	1.98	-----[b]

[a] The values of d_{av} are probably accurate to ± 5% absolute.

[b] The content of deuterium was not determined by ^{1}H NMR for this substrate.

shows, for example, that the reduction by D_2 of a mixture of 90% **1** and 10% **2** produces norbornanes containing 1.1 D in the *exo* position, and 0.1 D in the *endo* position. The observation that the incorporation of deuterium into the *endo* position does not exceed 0.1 D suggests that excess deuterium does not result from β incorporation into *endo*-2-norbornyl* (*endo**) moieties, nor from α incorporation into *exo*-2-norbornyl* (*exo**) moieties. On the other hand, β incorporation[73] of deuterium into *ca* 20% of the *exo** moieties adequately accounts for the excess deuterium, and is qualitatively consistent with the isotopic distributions shown in Figure 4 (*ca* 70% norbornane-d_1 and 20% norbornane-d_2) for the reduction of this mixture. Scheme III summarizes these proposals.

In the reduction of samples containing 97% **2** and 3% **1**, the presence of 3% **1** is not significant, and will not be considered in the discussion here. The total amount of deuterium incorporated into the norbornanes (1.4 D) is described by eq 4: 1.0 D in the *endo* position, and 0.4 D in the *exo* position. Since the incorporation of deuterium into the *endo* position does not exceed 1.0 D, the excess deuterium probably does not result from β incorporation of deuterium into the *endo** moieties. Scheme IV summarizes the proposed reactions occurring in the reduction of *endo**. The incorporation of excess deuterium into *endo** moieties probably occurs via α incorporation[74,75] and epimerization of *ca* 35% of the *endo** moieties to *exo** moieties followed by β incorporation of deuterium into *ca* 20% of the subsequent *exo** moieties.[76] The reactions shown in Scheme IV rationalize the incorporation of excess deuterium, and are in qualitative agreement with the isotopic distributions shown in Figure 4 (e.g. *ca* 60% norbornane-d_1, 20% norbornane-d_2, and 10% norbornane-d_3) for the reduction of 97% **2** and 3% **1**.

Scheme III. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 90% **1** and 10% **2**. Hydrogen atoms have been omitted for clarity.

D (1.1)

D (0.1)

D (1.1)

D (0.1)

+

D*

D*

D (0.2)
*(0.9)

*(0.1)

+

β-elim

D*

*(0.90)

*(0.10)

Scheme IV. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 97% **2** and 3% **1**. Hydrogen atoms have been omitted for clarity.

D (0.4)

D (1.0)

D (0.40)

D (0.35)

D (0.65)

+

β-elim

D*

D*

* (0.35)

D (0.35)

* (0.65)

+

α-elim

D*

* (0.03)

* (0.97)
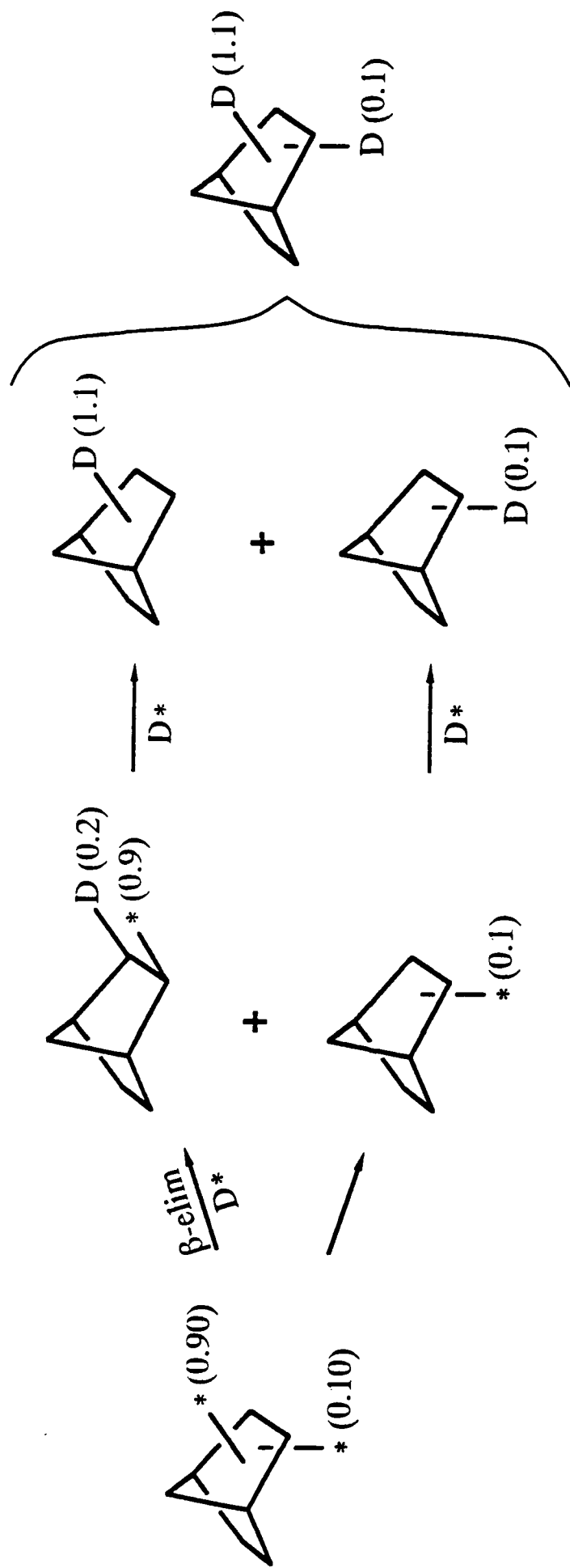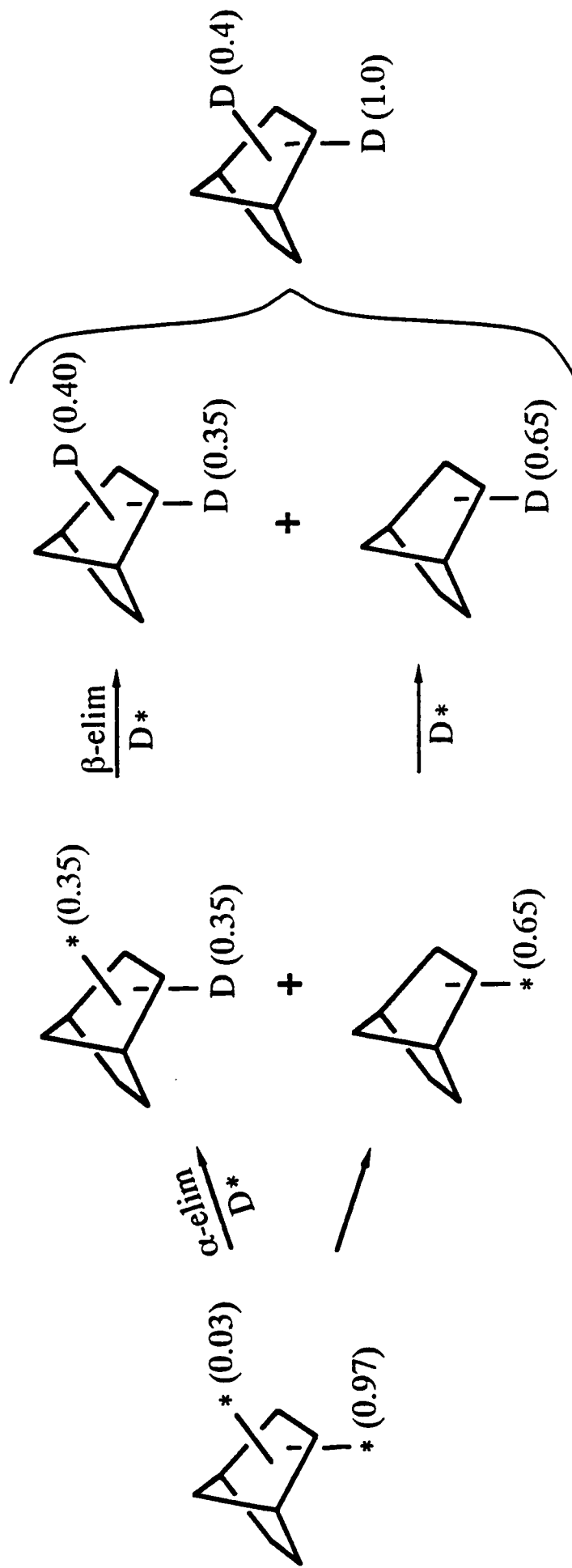
In summary, the reduction of exo* moieties occurs via (1) simple reductive elimination (major pathway, ca 70%), and (2) β incorporation of deuterium prior to reductive elimination (minor pathway, ca 20%). Since α incorporation into exo* does not occur, α-H activation of exo* probably does not occur; hence, epimerization from exo* to endo* does not occur. The reduction of endo* moieties occurs via (1) simple reductive elimination (major pathway, ca 65%), and (2) α-H activation and epimerization to exo* followed by reduction of exo* as described above (minor pathway, ca 35%). Since β incorporation into endo* does not occur, β-H activation (β-H elimination) of endo* probably does not occur. The reasons for the differences in reactivity between exo* and endo* cannot be determined from the experimental data. In the following two paragraphs, we provide largely speculative rationalizations[77] for the apparent differences in reactivity between these surface moieties assuming that the rate-limiting step for production of norbornane is reductive elimination from the surface.[50-52]

The observation of α-H activation/epimerization of endo* to exo*, but not of exo* to endo*, can be rationalized on the usual basis of a steric preference for exo by large substituents (here the surface of platinum). The following argument assumes that the barriers to reductive elimination as norbornane are similar for exo* and endo*. A norbornyl group bonded endo to Pt* is probably energetically destabilized (relative to exo*) due to the unfavorable steric interactions between the distal endo hydrogens and the surface of platinum; consequently, the barrier to conversion of endo* to exo* is relatively low, and the rate of conversion of endo* to exo* is competitive with the rate of reductive elimination of endo-2-norbornyl groups from the surface. The transition states for conversion of endo* to exo* and exo* to endo* are probably the same. Since exo* is a more stable surface species than endo*, the barrier to conversion of exo* to endo* is relatively high, and the

rate of conversion of *exo** to *endo** is negligible in comparison to the rate of reductive elimination of *exo*-2-norbornyl groups from the surface.

The observation of β-H elimination in *exo** moieties, but not in *endo** moieties can also be rationalized assuming that that the barriers to reductive elimination as norbornane are similar for *exo** and *endo**. The formation of *endo*-norbornene* from *endo** is disfavored because *endo*-norbornene* is destabilized relative to *endo** due to increased steric repulsions between the distal *endo* hydrogens and the surface of platinum. Consequently, the energy of the transition state for β-H elimination in *endo** is high relative to that for reductive elimination of this moiety; the rate of β-H elimination is, therefore, negligible compared to the rate of reductive elimination. Formation of *exo*-norbornene* from *exo** is probably less sterically demanding than formation of *endo*-norbornene* from *endo**. As a consequence, the transition state for β-H elimination in *exo** is comparable in energy to that for reductive elimination of this species; hence, the rate of β-H elimination is competitive with that for reductive elimination.

Mass Spectra of the Neopentanes. Figure 5 shows the mass spectral data for neopentane-d_0, and the neopentanes produced in the reductions by D_2 of mixtures of 1 and 2. No M^+ ion is observed in the mass spectrum of neopentane: the base peak is the expected $(M\text{-}CH_3)^+$ ion. We are reluctant to infer detailed isotopic compositions of the neopentanes from these data since we do not know isotope effects for loss of, for example, CH_3 relative to CH_2D; we can, however, infer qualitatively that the neopentanes from the reduction of 1 and 2 are predominantly composed of neopentane-d_1, and that the same isotopic species is (are) produced from both 1 and 2.

Figure 5. Mass spectra of neopentane-d_0 (bottom), and the neopentanes produced from the reductions by D_2 of a mixture of 97% **2** and 3% **1** (middle), and 90% **1** and 10% **2** (top).
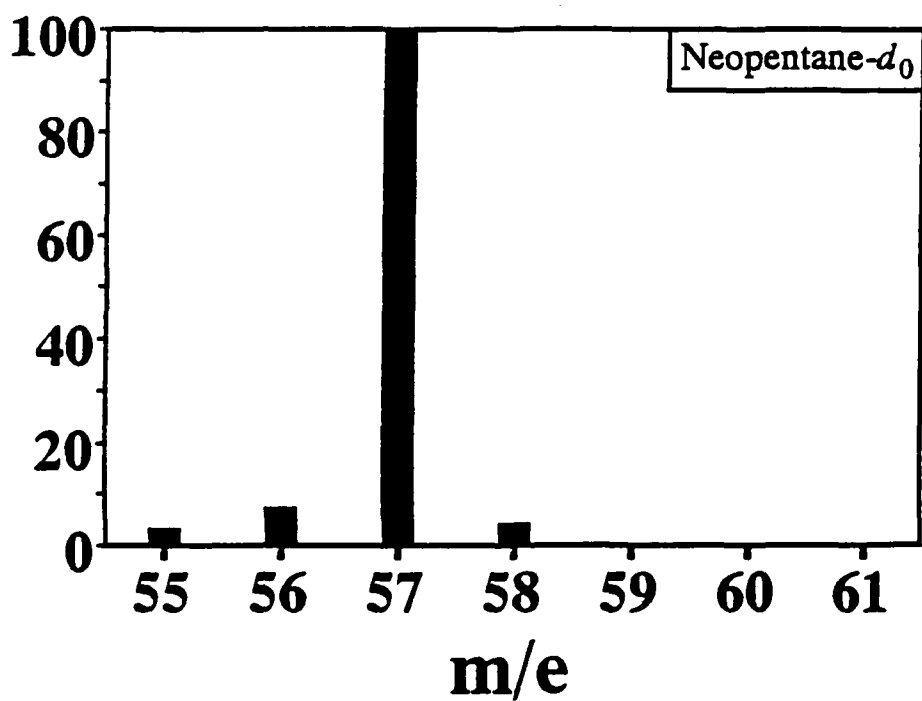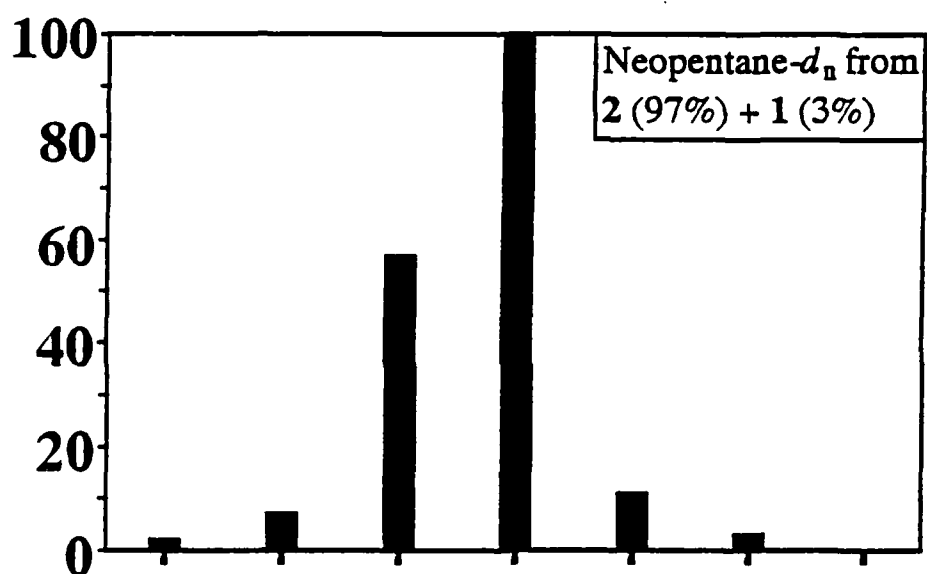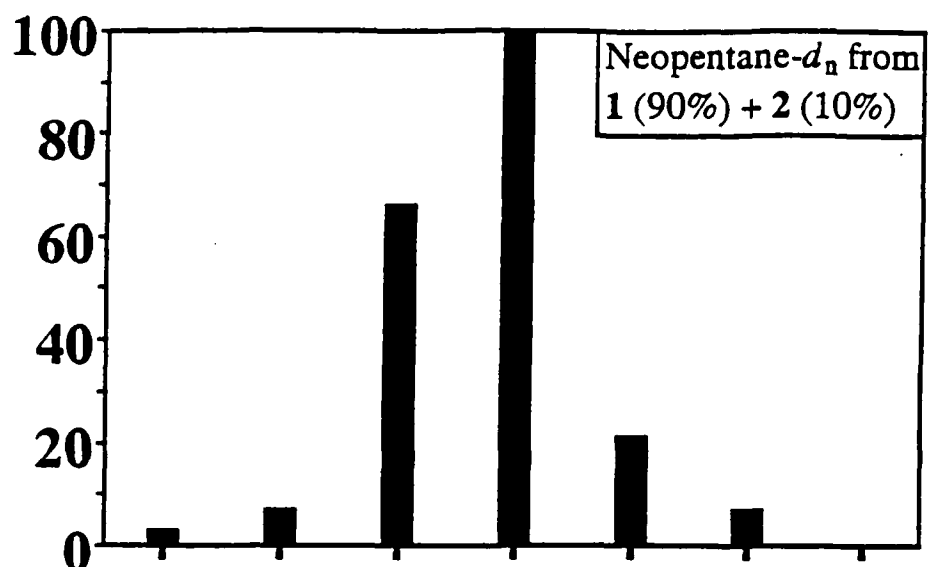
¹H NMR Spectra of the Homohypostrophanes. Figure 6 compares the ¹H NMR spectra of the homohypostrophanes (HOPH-d_n) produced in the reduction by H_2 of homohypostrophene, and the reductions by D_2 of homohypostrophene and samples containing **1** and **2**. The assignments of the ¹H resonances are described in the experimental section.[68,78] In the reduction by D_2 of homohypostrophene, the loss of the resonance attributed to the *exo* protons of HOPH indicates that deuterium adds exclusively to the *exo* positions of HOPH. Analogous reductions of samples containing predominantly **1** and predominantly **2** are indistiguishable from one another, and less isotopically clean than reduction of homohypostrophene. In the reductions of **1** and **2**, the loss of the resonance attributed to the *endo* protons of HOPH predominates: *ca* 3.1 H are lost from the *endo* positions and 2.1 H are lost from the *exo* positions. This observation indicates that deuterium adds predominantly to the *endo* positions of the HOP moiety originally coordinated to platinum in **1** and **2**.

Qualitatively, these data suggest that the reduction of coordinated homohypostrophene proceeds with stereochemistry that is predominantly opposite to that of the reduction of free homohypostrophene. The observation that deuterium is incorporated predominantly into the faces of the olefins in homohypostrophene that were coordinated to the platinum atoms in **1** and **2** indicates that adsorption on the surface occurs through initial attachment of the platinum atoms of these complexes. These results provide further support for our proposal that the mechanism of reduction of DOPtR₂ proceeds via initial association of the platinum atom with the surface of the catalyst: we observed previously that the reduction of coodinated norbornadiene (NBD) in NBDPtMe₂ proceeds with stereochemistry that is predominantly opposite to that of the reduction of free NBD.[51] Taken together, these results argue that the adsorption of the diolefin

Figure 6. ^{1}H NMR spectra (CDCl$_3$, 500 MHz) of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]-undecanes (homohypostrophanes) from: (i) the reduction by H$_2$ of homohypostrophene,[68] the reduction by D$_2$ of (ii) homohypostrophene, (iii) samples containing 97% **2** and 3% **1**, and (iv) samples containing 90% **1** and 10% **2**. The changes in chemical shifts between (i)-(iv) are probably due to isotope effects.[78]
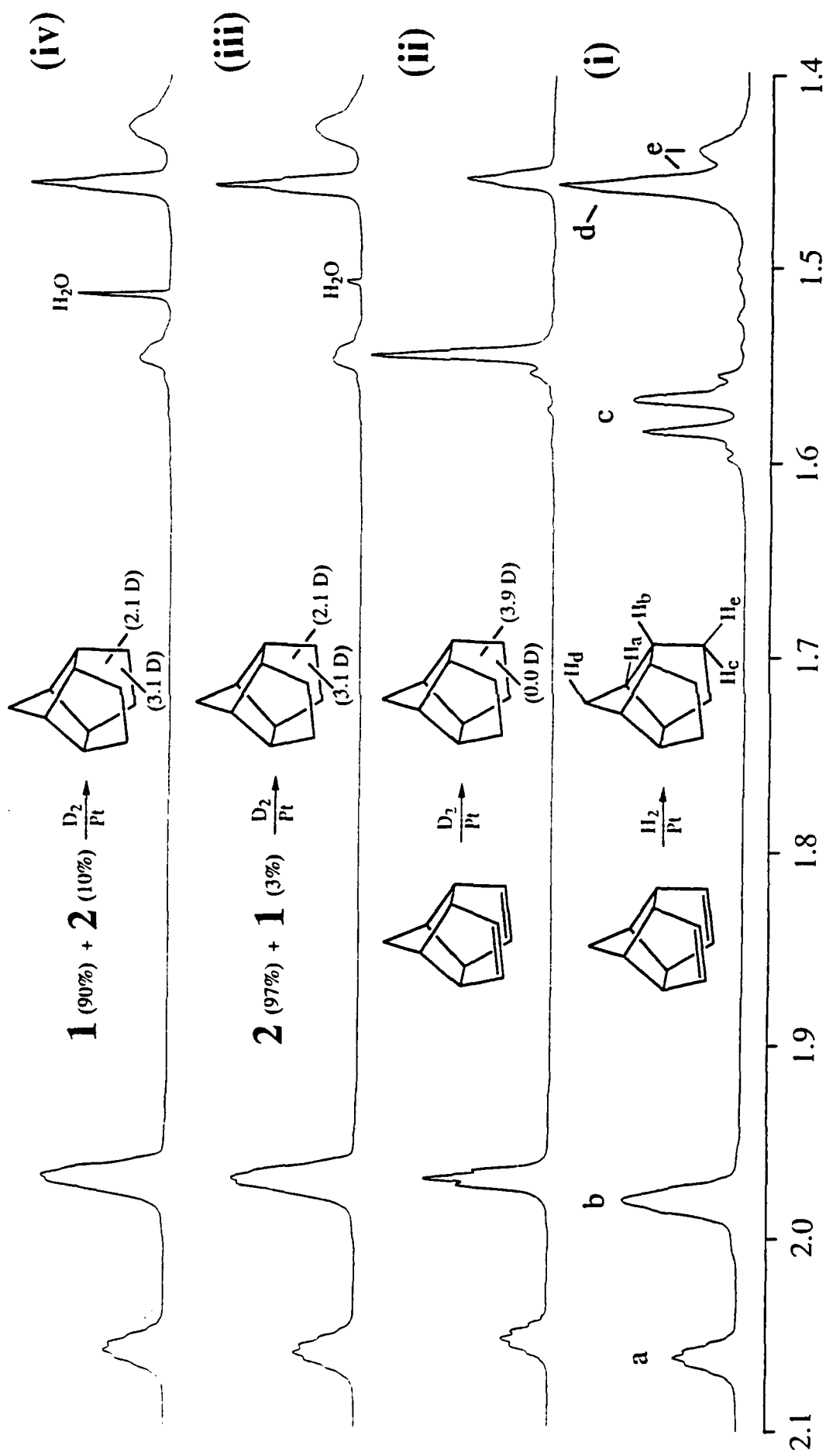
moieties of DOPtR$_2$ on the surface of the catalyst proceeds with retention of configuration, and are consistent with our assumption that the adsorption of the norbornyl moieties from 1 and 2 proceeds with retention of configuration.

Mass Spectral Data for the Homohypostrophanes. Figure 7 provides the mass spectral data for the homohypostrophanes produced in the reductions by D$_2$ of homohypostrophene and mixtures of 1 and 2. In all cases the major isotopomer produced is HOPH-d_4. The reduction of homohypostrophene produces HOPH-d_4 relatively cleanly (> 60%). Reductions of 1 and 2, however, produce significant quantities of other isotopomers, HOPH-d_n (n = 5-8).

The broader distribution of isotopomers of HOPH produced from the reductions of 1 and 2 relative to that produced from the reduction of homo-hypostrophene probably results from steric destabilization of the *endo*, *endo* bound surface diolefin, or the *endo*-surface alkyl. This additional strain energy probably allows the rate of other processes (e.g. isomerization of *endo*-HOP* to *exo*-HOP*) to become competitive with the rate of reductive elimination from the surface.

Figure 7. Isotopic distributions determined from the mass spectra of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecanes (homohypostrophanes) produced in the reductions by D$_2$ of homohypostrophene (bottom), a mixture of 97% **2** & 3% **1** (middle), and a mixture of 90% **1** & 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Figure showing three histograms of Percent Composition of Isotopomers versus Number of Deuteriums.

Top panel: HOPH-d_n from 1 (90%) + 2 (10%), $d_{av} = 5.22$

Middle panel: HOPH-d_n from 2 (97%) + 1 (3%), $d_{av} = 5.24$

Bottom panel: HOPH-d_n from Homohypostrophene, $d_{av} = 3.88$
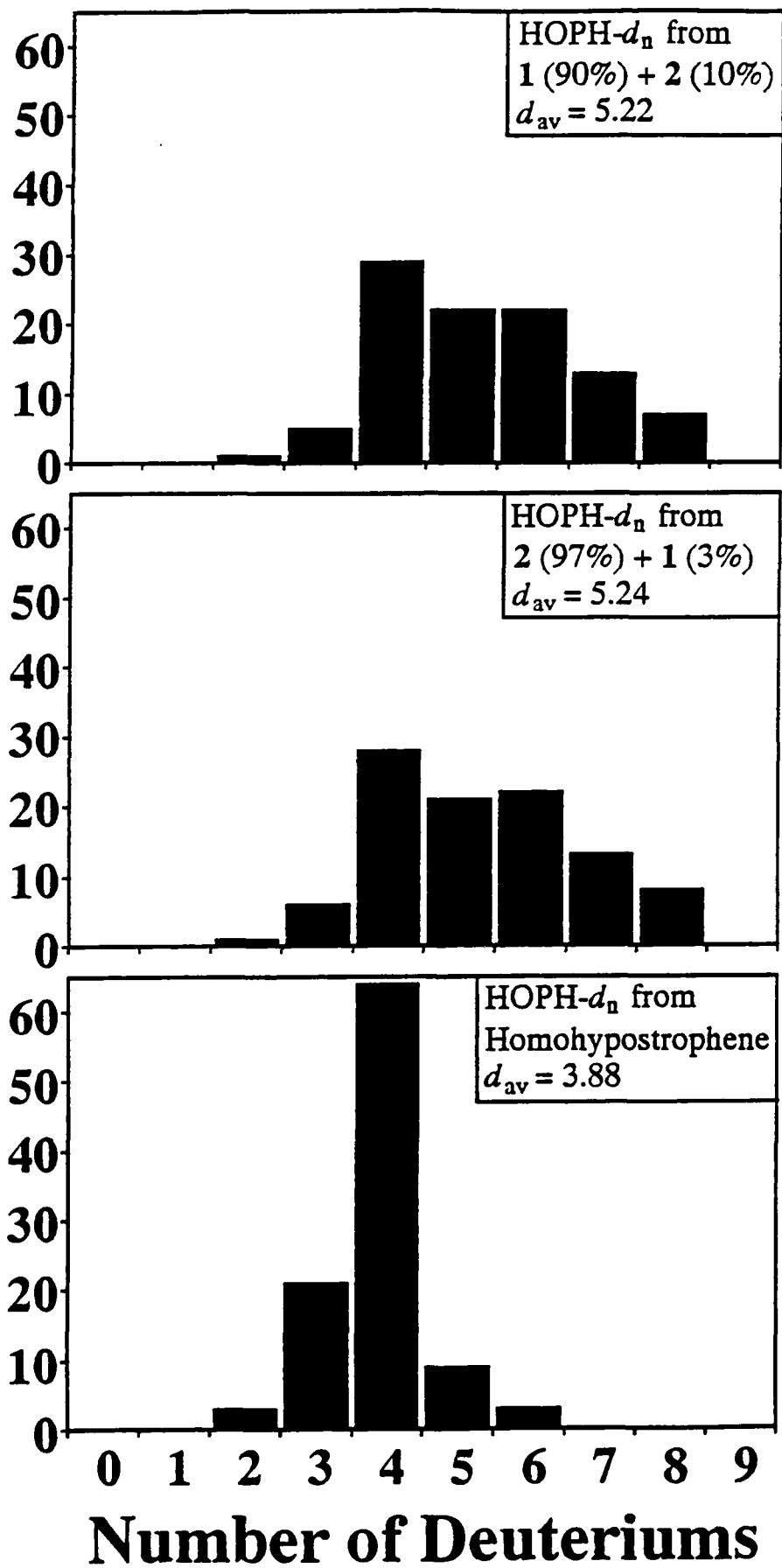
Conclusions

The major conclusions from this work are:

1. *The stereochemistry of the reduction of C* bonds by H* (D*) proceeds with retention of configuration.* This conclusion is based on the observation that the reduction of 1 with D_2 incorporates deuterium into the *exo* position of norbornane, and that the reduction of 2 with D_2 incorporates deuterium predominantly into the *endo* position of norbornane. Our proposed mechanisms for the reductions of exo-*2-norbornyl** and endo-*2-norbornyl** moieties (Schemes III and IV) argue that final reductive elimination of C* bonds from the surface proceeds with absolute retention of configuration. These arguments rely, however, on the correctness of the assumption that the adsorptions of 1 and 2 to form 2-norbornyl* moieties proceeds without loss of the stereochemistry of bonding between the 2-norbornyl moieties and platinum(II). Support for this assumption is detailed in the **Results and Discussion** section of this paper.

2. *The reduction of* exo-*2-norbornyl** *moieties is relatively clean; the reduction probably proceeds via simple reductive elimination from the surface (major pathway, ca 70%), and β-H elimination and incorporation of deuterium prior to reductive elimination from the surface (minor pathway, ca 20%).* The results which support this conclusion are (i) the incorporation of excess deuterium into the *exo* position of norbornane in the reduction by D_2 of a sample containing 90% 1 and 10% 2, and (ii) the distribution of isotopomers of norbornane produced from this reduction.

3. *The reduction of* endo-*2-norbornyl** *moieties is less straightforward; the reduction probably proceeds via simple reductive elimination from the surface*

(major pathway. ca *65%), and* α*-H activation and epimerization to* exo-2-*norbornyl* moieties followed by the reduction of these species as described in conclusion 2 (minor pathway,* ca *35%).* This conclusion is supported by (i) the incorporation of excess deuterium into the *exo* position of norbornane in the reduction by D_2 of a sample containing 97% **2** and 3% **1**, and (ii) the distribution of isotopomers of norbornane produced in this reaction.

The differences in reactivity between *exo** moieties and *endo** moieties probably result from greater steric destabilization of *endo** than of *exo**. Repulsions between the surface of platinum and the *endo* hydrogens of the *endo** moieties are responsible for this additional destabilization.

4. *The reduction of DOPtR$_2$ complexes occurs via initial adsorption of the platinum atom in the organometallic complex.* Support for this conclusion is the observation that the reductions by D_2 of **1** and **2** incorporate deuterium predominantly into the *endo* positions of homohypostrophane, but the reduction of homohypostrophene by D_2 incorporates deuterium exclusively into the *exo* positions of homohypostrophane.

Supplementary Material. We provide the following for **1** and **2**: procedures for the determination of structure, summary of the crystallographic data, atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms, complete tables of bond distances and angles, anisotropic displacement parameters for non-hydrogen atoms, coordinates for hydrogen atoms, observed and calculated structure factors, packing diagrams, and UV absorption spectra (44 pages). Ordering information is given on any current masthead page.

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(78) The changes in the chemical shifts of HOPH upon incorporation of deuterium are not due to concentration effects: a mixture (*ca* 1:1) of HOPH-d_0 and HOPH-d_4 (from the reduction by D_2 of homohypostrophene) gave chemical shifts consistent with those shown in Figure 7.

Captions for Figures and Schemes (cont)

Figure 6. ^{1}H NMR spectra (CDCl$_3$, 500 MHz) of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]-undecanes (homohypostrophanes) from: (i) the reduction by H$_2$ of homohypostrophene,[68] the reduction by D$_2$ of (ii) homohypostrophene, (iii) samples containing 97% **2** and 3% **1**, and (iv) samples containing 90% **1** and 10% **2**. The changes in chemical shifts between (i)-(iv) are probably due to isotope effects.[78]

Figure 7. Isotopic distributions determined from the mass spectra of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecanes (homohypostrophanes) produced in the reductions by D$_2$ of homohypostrophene (bottom), a mixture of 97% **2** & 3% **1** (middle), and a mixture of 90% **1** & 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Scheme I. Proposed Analogy Between Surface Alkyls Derived from Norbornene (left) and Those Derived from (Homohypostophene)neopentyl(*exo*-2-norbornyl)platinum(II) (right). HOP = Homohypostrophene; Np = Neopentyl.

Scheme II. Synthesis of Homohypostrophene.

Scheme III. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D$_2$ of a Mixture of 90% **1** and 10% **2**. Hydrogen atoms have been omitted for clarity.

Scheme IV. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D$_2$ of a Mixture of 97% **2** and 3% **1**. Hydrogen atoms have been omitted for clarity.

Captions for Figures and Schemes

Figure 1. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1**. The molecule crystallizes in a non-centrosymmetric space group; the absolute configuration (*R*) was determined crystallographically. The data for this structure were collected at 0 °C.

Figure 2. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**. The molecule crystallizes in a centrosymmetric space group; the *S* enantiomer is shown. The data for this structure were collected at -58 °C.

Figure 3. ^{1}H NMR spectra (C_6D_6, 500 MHz) of (i) norbornane-d_0, and the norbornanes from the reduction by D_2 of samples containing (ii) 97% **2** and 3% **1**, and (iii) 90% **1** and 10% **2**. ^{2}H NMR spectra (C_6D_6, 46.03 MHz) of the norbornanes from the reductions by D_2 of samples containing (iv) 97% **2** and 3% **1**, and (v) 90% **1** and 10% **2**.

Figure 4. Isotopic distributions determined from the mass spectra of the norbornanes produced from the reductions by D_2 of norbornene (bottom), a mixture of 97% **2** and 3% **1** (middle), and a mixture of 90% **1** and 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Figure 5. Mass spectra of neopentane-d_0 (bottom), and the neopentanes produced from the reductions by D_2 of a mixture of 97% **2** and 3% **1** (middle), and 90% **1** and 10% **2** (top).

Supplementary Material

for

The Reduction of C* Bonds Proceeds with Retention of Configuration: Stereochemical Investigation of the Heterogeneous Reduction by Dideuterium of (Homohypostrophene)neopentyl(2-norbornyl)platinum(II) Complexes on Platinum Black.

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Supplementary Material.   We provide the following for **1** and **2**: procedures for the determination of structure, summary of the crystallographic data, atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms, complete tables of bond distances and angles, anisotropic displacement parameters for non-hydrogen atoms, coordinates for hydrogen atoms, observed and calculated structure factors, packing diagrams, and UV absorption spectra (44 pages).

X-ray Crystallography: General. We obtained the data using a Nicolet R3m/V four-circle diffractometer equipped with a LT-1 low-temperature device. Collection of the data was controlled with the Nicolet P3 program.[1] We checked the symmetry of the unit cells with the program XCELL, and processed the raw data with the program XDISK. We used the program XEMP to perform empirical absorption corrections, and solved the structures by use of the SHELXTL-PLUS[2] package of programs. Drawings were produced using the Nicolet program XP.

X-ray Crystallography for (Homohypostrophene)neopentyl-(*exo*norbornyl)platinum(II) *1.* We grew crystalline plates of **1** by low temperature crystallization from hexane/ethanol. The plates were transparent except for a cloudy line along one of the diagonals. We cut one of the plates (0.1 x 0.2 x 0.3 mm) along the diagonal, and mounted it in air as follows. We attached the crystal to a 0.30 mm glass fiber with a minimum amount of epoxy glue. We glued the fiber to a 1/8" diameter brass pin using epoxy, and attached the pin to the goniometer head. We transferred the goniometer head to the diffractometer where the crystal was bathed by a cold nitrogen stream (0 (1) °C).

We used the data from a random search of reciprical space to index the unit cell. A lattice determination using both the P3 program and XCELL suggested a primitive orthorhombic cell. Examination of the axial photographs confirmed this assignment. We obtained the final unit cell parameters by performing a least squares refinement of 48 selected reflections, including four Friedel pairs, in the range $15° < 2\theta < 30°$.

We collected a total of 4005 reflections in the range $4° < 2\theta < 48°$ (-h, -1, l to h, k, l). Of these, 3001 were unique reflections, and 1791 with $F_0 > 6\sigma(F_0)$ were used in the structure solution. We measured the intensities of three check

reflections, (1, -2, -8), (-3, -3, 1) and (0, -5, 1), after every 60 reflections. These check reflections showed that the crystal did not decay during the 52 hours of exposure.

Systematic absences uniquely determined the space group to be $P2_12_12_1$; successful solution in this space group confirmed its choice. We located the platinum atom from direct methods, and used standard difference map techniques to find the remaining non-hydrogen atoms. After all of the non-hydrogen atoms were located and refined anisotropically, a difference map revealed approximately one-half of the hydrogen atom positions. The hydrogen atoms were placed in calculated positions (Uiso(H) = 1.2Uiso(C); dC-H = 0.96 Å) for refinement. We performed the refinement to convergence (Δ/σ(max) < 0.010) with this model. The weighting scheme was w = $[\sigma^2(F) + gF^2]^{-1}$ (g= 1.5 x 10^{-4}). The final difference map contained three peaks (1.61, 1.44 and 1.41 eÅ-3) located near the platinum atom. All other peaks were less than 0.67 eÅ-3.

The molecule crystallizes chirally, with one unique crystallographically independent molecule. We determined the correctness of the enantiomorph by inversion of configuration. With the program XEMP, we applied a semi-empirical absorption correction using scans near χ = 270, and scans from 5 reflections in the range 14° < 2θ < 31°. Refinement yielded a structure with no non-positive definite atoms.

Table Ia. Summary of the crystallographic data for **1**

Color of crystal	yellow
Empirical formula	$C_{23}H_{34}Pt$
Crystal dimensions, mm	0.10 x 0.20 x 0.30
Space group	$P2_12_12_1$ (No. 19)
Cell dimensions	
a, Å	9.980 (4)
b, Å	11.419 (4)
c, Å	16.912 (3)
Temperature, °C	0 (1)
Wavelength, Å	0.71073
Z (molecules/cell)	4
Volume, Å^3	1927 (1)
d_{calcd}, g cm^{-3}	1.74
Linear absorption coefficient, cm^{-1}	73.6
Scan type	$\theta - 2\theta$
Scan speed, deg/min	3 - 30
Scan width, deg (+ dispersion)	2.0
Background / scan ratio	0.50
2θ range, deg	4 - 48
Data collected	$h, \pm k, l$
$F(000)$	1000
Parameters refined	217
Total number of reflections collected	4005
Number of unique reflections	3001
R_{int}	0.0524
number with $F_o > 6.00\sigma(F_o)$	1791
$R(F)$	0.0548
$R_w(F)$	0.0547
"Goodness of fit" for last cycle	1.099
Largest Δ/σ for last cycle	0.002
Final difference map (maximum)	1.61, 1.44 and 1.41 eÅ^{-3} near Pt
(remainder)	<0.67 eÅ^{-3}

[a] $R = \Sigma|(Fo\text{-}Fc)|/\Sigma Fo$

[b] $R_w = \Sigma(w^{1/2}|(Fo\text{-}Fc)|/\Sigma(w^{1/2}Fo)$, $w = [\sigma^2(F) + gF^2]^{-1}$

Table Ib. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 1[a]

atom	x	y	z	U(eq)
Pt	6775(1)	10325(1)	5380(1)	44(1)
C(1)	10982(20)	8135(23)	5535(15)	52(9)
C(2)	10388(22)	8914(25)	4924(16)	50(10)
C(3)	9802(22)	8365(30)	6093(14)	55(11)
C(4)	8899(20)	8428(21)	4789(13)	32(8)
C(5)	8484(19)	8043(25)	5600(13)	48(10)
C(6)	9563(21)	9672(29)	6170(13)	46(8)
C(7)	10016(19)	10074(22)	5316(17)	49(10)
C(8)	8770(20)	10481(23)	4801(12)	37(6)
C(9)	8148(23)	9511(18)	4475(11)	36(7)
C(10)	7403(24)	8759(24)	6044(13)	37(8)
C(11)	7997(22)	9668(32)	6342(13)	59(10)
C(12)	5145(25)	10811(41)	6107(15)	119(21)
C(13)	4513(35)	9969(43)	6689(22)	130(23)
C(14)	3150(43)	10521(58)	6832(30)	204(34)
C(15)	3392(34)	11517(45)	7320(20)	121(20)
C(16)	4950(61)	11544(47)	7432(20)	142(27)
C(17)	5551(47)	11806(36)	6736(20)	118(20)
C(18)	5088(38)	10313(51)	7428(22)	127(23)
C(19)	5923(22)	11407(23)	4532(17)	51(10)
C(20)	4785(28)	11055(27)	3964(17)	59(11)
C(21)	5196(30)	10022(23)	3435(14)	68(14)
C(22)	4496(37)	12091(31)	3433(17)	89(16)
C(23)	3457(24)	10758(31)	4368(17)	89(15)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table Ic. Complete listing of bond lengths (Å) for **1**

Pt-C(8)	2.226 (20)	C(6)-C(11)	1.590 (30)
Pt-C(9)	2.256 (21)	C(7)-C(8)	1.589 (30)
Pt-C(10)	2.202 (25)	C(8)-C(9)	1.384 (32)
Pt-C(11)	2.167 (24)	C(10)-C(11)	1.297 (41)
Pt-C(12)	2.112 (27)	C(12)-C(13)	1.514 (55)
Pt-C(19)	2.075 (27)	C(12)-C(17)	1.609 (54)
C(1)-C(2)	1.487 (37)	C(13)-C(14)	1.519 (60)
C(1)-C(3)	1.533 (32)	C(13)-C(18)	1.430 (54)
C(2)-C(4)	1.602 (31)	C(14)-C(15)	1.426 (76)
C(2)-C(7)	1.527 (38)	C(15)-C(16)	1.566 (69)
C(3)-C(5)	1.601 (31)	C(16)-C(17)	1.355 (56)
C(3)-C(6)	1.517 (47)	C(16)-C(18)	1.412 (79)
C(4)-C(5)	1.500 (32)	C(19)-C(20)	1.542 (38)
C(4)-C(9)	1.541 (31)	C(20)-C(21)	1.536 (39)
C(5)-C(10)	1.548 (33)	C(20)-C(22)	1.513 (45)
C(6)-C(7)	1.581 (37)	C(20)-C(23)	1.529 (38)

Table Id. Complete listing of bond angles (°) for **1**

C(8)-Pt-C(9)	36.0(8)	C(3)-C(5)-C(10)	101.5(19)
C(8)-Pt-C(10)	92.0(9)	C(4)-C(5)-C(10)	118.8(21)
C(9)-Pt-C(10)	80.7(8)	C(3)-C(6)-C(7)	99.3(19)
C(8)-Pt-C(11)	81.7(8)	C(3)-C(6)-C(11)	99.6(23)
C(9)-Pt-C(11)	91.4(9)	C(7)-C(6)-C(11)	116.7(18)
C(10)-Pt-C(11)	34.5(11)	C(2)-C(7)-C(6)	102.4(21)
C(8)-Pt-C(12)	157.5(13)	C(2)-C(7)-C(8)	101.9(20)
C(9)-Pt-C(12)	166.1(12)	C(6)-C(7)-C(8)	111.2(16)
C(10)-Pt-C(12)	97.8(12)	Pt-C(8)-C(7)	115.8(14)
C(11)-Pt-C(12)	95.0(10)	Pt-C(8)-C(9)	73.2(13)
C(8)-Pt-C(19)	90.8(9)	C(7)-C(8)-C(9)	109.6(21)
C(9)-Pt-C(19)	91.4(9)	Pt-C(9)-C(4)	113.1(13)
C(10)-Pt-C(19)	162.2(10)	Pt-C(9)-C(8)	70.8(12)
C(11)-Pt-C(19)	162.8(11)	C(4)-C(9)-C(8)	106.6(18)
C(12)-Pt-C(19)	86.0(12)	Pt-C(10)-C(5)	112.4(15)
C(2)-C(1)-C(3)	91.1(18)	Pt-C(10)-C(11)	71.2(17)
C(1)-C(2)-C(4)	105.2(20)	C(5)-C(10)-C(11)	107.0(21)
C(1)-C(2)-C(7)	108.3(22)	Pt-C(11)-C(6)	114.5(16)
C(4)-C(2)-C(7)	97.9(17)	Pt-C(11)-C(10)	74.2(15)
C(1)-C(3)-C(5)	105.7(18)	C(6)-C(11)-C(10)	112.4(25)
C(1)-C(3)-C(6)	110.0(22)	Pt-C(12)-C(13)	122.2(30)
C(5)-C(3)-C(6)	98.1(19)	Pt-C(12)-C(17)	112.1(22)
C(2)-C(4)-C(5)	103.2(18)	C(13)-C(12)-C(17)	97.1(26)
C(2)-C(4)-C(9)	102.8(18)	C(12)-C(13)-C(14)	102.3(37)
C(5)-C(4)-C(9)	114.6(18)	C(12)-C(13)-C(18)	103.1(35)
C(3)-C(5)-C(4)	100.4(17)	C(14)-C(13)-C(18)	96.1(35)
C(13)-C(14)-C(15)	105.7(36)	Pt-C(19)-C(20)	125.3(19)
C(14)-C(15)-C(16)	104.7(35)	C(19)-C(20)-C(21)	111.5(22)
C(15)-C(16)-C(17)	109.8(36)	C(19)-C(20)-C(22)	107.8(25)
C(15)-C(16)-C(18)	94.4(41)	C(21)-C(20)-C(22)	107.8(23)
C(17)-C(16)-C(18)	99.9(38)	C(19)-C(20)-C(23)	114.7(23)
C(12)-C(17)-C(16)	107.9(37)	C(21)-C(20)-C(23)	108.7(25)
C(13)-C(18)-C(16)	103.7(38)	C(22)-C(20)-C(23)	105.9(25)

Table Ie. Anisotropic displacement parameters ($\mathrm{\AA}^2 \times 10^3$) for **1**[a]

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pt	26(1)	75(1)	30(1)	8(1)	0(1)	-5(1)
C(1)	25(11)	63(13)	69(19)	15(12)	23(13)	-22(15)
C(2)	19(11)	64(21)	67(18)	1(14)	15(12)	-26(17)
C(3)	27(13)	113(29)	24(13)	38(16)	-6(12)	-5(15)
C(4)	25(10)	28(15)	42(15)	12(10)	8(11)	-8(12)
C(5)	18(13)	72(21)	55(16)	-4(11)	0(11)	7(13)
C(6)	39(12)	53(18)	46(13)	3(17)	-16(11)	-10(18)
C(7)	22(10)	48(24)	78(17)	2(10)	14(14)	5(18)
C(8)	36(10)	41(9)	34(13)	-3(12)	14(9)	-15(13)
C(9)	45(11)	26(13)	37(11)	20(16)	-3(13)	-20(12)
C(10)	46(13)	33(17)	32(12)	-1(13)	3(12)	-3(13)
C(11)	38(14)	101(24)	38(12)	23(20)	4(12)	-17(17)
C(12)	32(15)	297(61)	28(14)	43(24)	-19(14)	-45(25)
C(13)	92(25)	177(57)	122(31)	-22(30)	95(26)	-30(33)
C(14)	80(27)	387(91)	144(37)	-3(55)	28(31)	-106(52)
C(15)	51(22)	236(53)	75(22)	83(31)	-13(20)	-39(28)
C(16)	249(65)	128(43)	49(22)	-85(46)	40(31)	-62(26)
C(17)	154(38)	130(40)	70(25)	-19(32)	33(25)	-47(26)
C(18)	96(28)	200(56)	85(26)	-19(36)	-31(22)	-69(35)
C(19)	41(13)	43(18)	70(18)	18(12)	14(16)	36(17)
C(20)	61(18)	50(21)	67(19)	0(17)	-11(17)	3(19)
C(21)	100(22)	62(34)	41(13)	18(18)	-35(15)	-20(15)
C(22)	106(28)	100(32)	61(20)	19(24)	-32(21)	8(21)
C(23)	35(17)	143(35)	88(21)	-19(17)	24(15)	-32(20)

[a] The anisotropic displacement exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \ldots + 2hka^*b^* U_{12})$.

Table If. Coordinates of hydrogen atoms (x 10^4) for **1**

atom	x	y	z
H(1A)	11827	8407	5734
H(1B)	11042	7333	5368
H(2A)	10916	8993	4452
H(3A)	9875	7968	6592
H(4A)	8893	7796	4415
H(5A)	8295	7219	5620
H(6A)	10082	10015	6588
H(7A)	10737	10630	5329
H(8A)	8907	11141	4458
H(9A)	7882	9523	3929
H(10A)	6743	8355	6354
H(11A)	7732	9878	6869
H(12A)	4463	11115	5764
H(13A)	4424	9163	6539
H(14A)	2752	10784	6347
H(14B)	2506	10046	7106
H(15A)	3171	12238	7058
H(15B)	2942	11491	7821
H(16A)	5287	11929	7896
H(17A)	6506	11865	6795
H(17B)	5199	12550	6574
H(18A)	4551	10015	7854
H(18B)	5993	10039	7485
H(19A)	5628	12088	4816
H(19B)	6653	11660	4204
H(21A)	6016	10204	3164
H(21B)	4508	9853	3055
H(21C)	5331	9352	3769
H(22A)	4225	12770	3728
H(22B)	3801	11881	3067
H(22C)	5304	12262	3147
H(23A)	3151	11392	4693
H(23B)	3612	10080	4691
H(23C)	2790	10580	3977

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.

H	K	L	10FO	10FC	10S
2	0	0	284	265	18
4	0	0	259	240	37
6	0	0	2121	2125	27
8	0	0	1181	1157	24
10	0	0	213	222	-114
1	1	0	3100	2914	24
2	1	0	402	399	14
3	1	0	525	489	25
4	1	0	414	382	17
5	1	0	1359	1257	14
6	1	0	90	156	-67
7	1	0	1603	1507	21
8	1	0	86	124	-61
9	1	0	680	695	23
10	1	0	233	178	-79
11	1	0	117	179	-87
0	2	0	3688	3535	111
1	2	0	2439	2428	119
2	2	0	1301	1264	24
3	2	0	849	876	22
4	2	0	159	70	-73
5	2	0	923	864	18
6	2	0	1820	1775	68
7	2	0	83	62	-59
8	2	0	1101	1101	18
9	2	0	388	380	33
10	2	0	190	193	-107
11	2	0	389	394	61
1	3	0	1737	1832	21
2	3	0	858	906	43
3	3	0	461	408	51
4	3	0	1042	1008	15
5	3	0	1084	1036	15
6	3	0	545	523	22
7	3	0	1414	1327	50
8	3	0	269	148	-50
9	3	0	516	540	30
10	3	0	543	573	28
11	3	0	156	221	-83
0	4	0	1565	1559	27
1	4	0	237	263	34
2	4	0	625	652	23
3	4	0	1402	1395	31
4	4	0	336	287	40
5	4	0	1387	1354	52
6	4	0	1159	1101	18
7	4	0	138	125	-98
8	4	0	733	695	23
9	4	0	630	649	47
10	4	0	97	44	-68
1	5	0	948	986	32
2	5	0	1050	1118	53
3	5	0	169	23	-89
4	5	0	1500	1490	25
5	5	0	617	613	21
6	5	0	791	742	45
7	5	0	858	826	20
8	5	0	455	419	29
9	5	0	116	96	-85
10	5	0	684	691	24
0	6	0	1005	1020	42
1	6	0	665	684	34
2	6	0	103	47	-70
3	6	0	1427	1483	20
4	6	0	158	220	-85
5	6	0	982	1007	55
6	6	0	452	414	42
7	6	0	145	66	-75
8	6	0	237	121	-80
9	6	0	716	634	48
10	6	0	103	15	-73
1	7	0	301	226	35
2	7	0	1040	1119	24
3	7	0	87	137	-62
4	7	0	1097	1073	51
5	7	0	212	77	-106
6	7	0	552	590	61
7	7	0	93	31	-66
8	7	0	463	462	33
9	7	0	99	20	-70
0	8	0	148	45	-78
1	8	0	500	516	21
2	8	0	225	200	-69
3	8	0	1021	1027	47
4	8	0	183	169	-87
5	8	0	670	605	26
6	8	0	111	12	-79
7	8	0	106	7	-77
8	8	0	191	114	-135
9	8	0	361	415	-65
1	9	0	256	283	-44
2	9	0	691	722	27
3	9	0	95	18	-67
4	9	0	642	553	63
5	9	0	145	53	-79
6	9	0	259	243	-82
7	9	0	221	213	-89
8	9	0	126	209	-93
0	10	0	377	296	62
1	10	0	305	257	42
2	10	0	324	309	-65
3	10	0	524	453	67
4	10	0	153	128	-79
5	10	0	374	345	-69
6	10	0	286	126	-62
7	10	0	118	20	-87
1	11	0	415	384	32
2	11	0	315	250	-54
3	11	0	116	32	-86
4	11	0	399	340	46
5	11	0	106	99	-106
6	11	0	118	67	-89
0	12	0	464	324	-88
1	12	0	213	130	-72
2	12	0	326	231	-70
3	12	0	104	180	-74
4	12	0	103	33	-73
1	13	0	247	221	-77
1	0	1	472	440	8
2	0	1	1464	1424	13
3	0	1	811	761	14
4	0	1	1899	1761	36
5	0	1	474	461	18
6	0	1	834	779	15
7	0	1	82	40	-58
8	0	1	549	528	24
9	0	1	213	255	-76
10	0	1	1010	1049	26
11	0	1	325	217	49
-11	1	1	761	816	22
-10	1	1	263	222	-113
-9	1	1	977	1034	18
-8	1	1	392	330	31
-7	1	1	190	81	-79
-6	1	1	342	369	27
-5	1	1	1540	1505	18
-4	1	1	521	473	23
-3	1	1	2425	2245	29
-2	1	1	341	318	20
-1	1	1	1108	963	30
0	1	1	338	304	12
1	1	1	1079	1023	15
2	1	1	309	295	19
3	1	1	2329	2316	22
4	1	1	493	489	16
5	1	1	1498	1470	19
6	1	1	387	326	23
7	1	1	236	85	-71
8	1	1	340	343	35
9	1	1	1001	1023	18
10	1	1	257	227	-63
11	1	1	758	804	23
-11	2	1	330	196	-65
-10	2	1	835	867	32
-9	2	1	412	412	52
-8	2	1	711	713	20
-7	2	1	523	534	43
-6	2	1	661	604	17
-5	2	1	486	472	23
-4	2	1	1922	1787	24
-3	2	1	540	559	17
-2	2	1	1554	1434	66
-1	2	1	647	676	26
0	2	1	263	296	18
1	2	1	621	602	40
2	2	1	1491	1448	40
3	2	1	572	471	19
4	2	1	1887	1795	67
5	2	1	527	485	20
6	2	1	630	637	23
7	2	1	547	529	30
8	2	1	695	705	22
9	2	1	385	422	36
10	2	1	819	861	25
11	2	1	164	211	-83
-11	3	1	487	576	34
-10	3	1	320	253	47
-9	3	1	696	735	21
-8	3	1	553	593	26
-7	3	1	298	277	40
-6	3	1	921	936	40
-5	3	1	997	915	15
-4	3	1	784	781	15
-3	3	1	1804	1748	43
-2	3	1	759	753	30
-1	3	1	821	823	25
0	3	1	1491	1583	35
1	3	1	791	871	29
2	3	1	773	787	16
3	3	1	1765	1750	30
4	3	1	793	756	14
5	3	1	962	951	41
6	3	1	922	892	22
7	3	1	273	288	-48
8	3	1	594	561	26
9	3	1	683	755	21
10	3	1	123	254	-88
11	3	1	517	574	46
-10	4	1	574	602	31
-9	4	1	263	354	-76
-8	4	1	517	452	26
-7	4	1	908	903	21
-6	4	1	370	332	38
-5	4	1	794	795	55
-4	4	1	1269	1194	17
-3	4	1	373	380	-72
-2	4	1	845	897	19
-1	4	1	1106	1199	37
0	4	1	378	383	20
1	4	1	1137	1244	54
2	4	1	838	893	19
3	4	1	351	353	25
4	4	1	1271	1180	28
5	4	1	776	770	19
6	4	1	325	365	35
7	4	1	918	893	21
8	4	1	452	455	29
9	4	1	378	342	37
10	4	1	605	584	26
-10	5	1	141	223	-86
-9	5	1	466	478	63
-8	5	1	589	570	25
-7	5	1	283	259	-48
-6	5	1	1057	981	72
-5	5	1	490	481	25
-4	5	1	417	393	26
-3	5	1	793	797	48
-2	5	1	802	855	59
-1	5	1	450	464	20
0	5	1	1645	1725	38
1	5	1	382	408	19
2	5	1	782	867	21
3	5	1	773	777	18
4	5	1	433	382	36
5	5	1	503	500	44
6	5	1	953	960	21
7	5	1	305	224	43
8	5	1	595	599	49
9	5	1	451	493	37
10	5	1	323	211	52
-10	6	1	336	276	49
-9	6	1	522	455	30
-8	6	1	118	206	-87
-7	6	1	802	739	21
-6	6	1	324	214	-67
-5	6	1	733	711	29
-4	6	1	505	469	26
-3	6	1	400	369	29
-2	6	1	502	518	56
-1	6	1	1148	1191	51
0	6	1	281	315	30
1	6	1	1119	1191	32
2	6	1	452	510	25
3	6	1	363	371	30
4	6	1	477	450	36
5	6	1	727	700	59
6	6	1	235	224	-70
7	6	1	776	736	23
8	6	1	240	213	-111
9	6	1	439	440	38
10	6	1	231	269	-105
-9	7	1	275	222	-105
-8	7	1	673	575	45
-7	7	1	93	133	-66
-6	7	1	770	762	38
-5	7	1	222	161	-81
-4	7	1	256	216	-57
-3	7	1	318	316	39
-2	7	1	750	736	23
-1	7	1	237	280	-41
0	7	1	1073	1079	32
1	7	1	303	274	31
2	7	1	754	757	53
3	7	1	352	315	48
4	7	1	293	229	-55
5	7	1	221	182	-82
6	7	1	795	749	51
7	7	1	168	108	-92
8	7	1	631	584	27
9	7	1	195	217	-94

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
-9	8	1	332	292	55	-1	11	1	200	128	-59	-10	2	2	265	152	-78	1	4	2	742	762	25	-5	7	2	379	422	48
-8	8	1	100	158	-71	0	11	1	394	341	29	-9	2	2	423	423	42	2	4	2	1093	1118	23	-4	7	2	863	942	33
-7	8	1	643	591	58	1	11	1	145	118	-57	-8	2	2	1009	927	23	3	4	2	1085	1090	25	-3	7	2	433	433	40
-6	8	1	334	215	-68	2	11	1	174	170	-123	-7	2	2	643	573	27	4	4	2	592	653	28	-2	7	2	894	906	29
-5	8	1	601	577	29	3	11	1	272	208	-73	-6	2	2	1463	1475	22	5	4	2	1078	1147	26	-1	7	2	486	479	50
-4	8	1	94	58	-67	4	11	1	100	126	-71	-5	2	2	789	741	19	6	4	2	1015	1003	25	0	7	2	197	185	-75
-3	8	1	295	194	-62	5	11	1	281	180	-68	-4	2	2	468	429	22	7	4	2	408	400	47	1	7	2	495	493	28
-2	8	1	110	122	-79	6	11	1	272	273	-72	-3	2	2	928	948	20	8	4	2	597	565	36	2	7	2	842	922	32
-1	8	1	885	846	52	-4	12	1	131	195	-93	-2	2	2	1319	1321	24	9	4	2	647	673	36	3	7	2	393	459	55
0	8	1	258	280	-50	-3	12	1	142	62	-89	-1	2	2	1138	1151	29	10	4	2	101	244	-101	4	7	2	989	973	30
1	8	1	889	860	33	-2	12	1	302	156	-63	0	2	2	2246	2227	25	-10	5	2	517	598	44	5	7	2	440	412	50
2	8	1	146	159	-82	-1	12	1	250	194	-68	1	2	2	1127	1093	21	-9	5	2	195	222	-144	6	7	2	470	409	48
3	8	1	250	176	-86	0	12	1	191	19	-66	2	2	2	1313	1348	23	-8	5	2	334	414	54	7	7	2	99	31	-99
4	8	1	93	43	-66	1	12	1	164	192	-58	3	2	2	820	974	24	-7	5	2	624	633	31	8	7	2	243	387	-120
5	8	1	575	567	39	2	12	1	104	162	-104	4	2	2	426	478	31	-6	5	2	706	708	27	9	7	2	364	221	-69
6	8	1	129	201	-81	3	12	1	187	62	-87	5	2	2	713	701	23	-5	5	2	589	649	32	-8	8	2	220	151	-115
7	8	1	605	602	37	4	12	1	236	190	-96	6	2	2	1493	1437	24	-4	5	2	1139	1245	27	-7	8	2	188	73	-159
8	8	1	218	175	-114	-1	13	1	112	68	-57	7	2	2	630	585	30	-3	5	2	591	634	28	-6	8	2	93	185	-93
9	8	1	276	285	-76	0	13	1	106	104	-53	8	2	2	1006	1000	26	-2	5	2	823	869	26	-5	8	2	478	479	43
-8	9	1	214	376	-105	1	13	1	115	74	-59	9	2	2	476	438	41	-1	5	2	811	888	19	-4	8	2	355	403	56
-7	9	1	298	142	-74	0	0	2	3313	3267	14	10	2	2	236	131	-107	0	5	2	735	755	35	-3	8	2	888	886	31
-6	9	1	565	493	62	1	0	2	1559	1486	19	11	2	2	374	330	62	1	5	2	870	888	45	-2	8	2	340	328	56
-5	9	1	323	256	-76	2	0	2	1693	1601	17	-11	3	2	299	282	-74	2	5	2	796	836	26	-1	8	2	416	354	-75
-4	9	1	259	267	-62	3	0	2	396	316	26	-10	3	2	548	559	37	3	5	2	641	665	29	0	8	2	170	113	-120
-3	9	1	168	93	-80	4	0	2	1096	1007	19	-9	3	2	508	505	37	4	5	2	1203	1230	24	1	8	2	388	371	39
-2	9	1	442	399	35	5	0	2	369	369	31	-8	3	2	459	495	39	5	5	2	710	661	29	2	8	2	380	362	53
-1	9	1	207	92	-62	6	0	2	1523	1429	21	-7	3	2	1176	1107	24	6	5	2	746	717	30	3	8	2	878	918	32
0	9	1	801	742	32	7	0	2	1030	976	22	-6	3	2	657	659	26	7	5	2	604	612	38	4	8	2	439	364	51
1	9	1	230	126	-52	8	0	2	951	973	24	-5	3	2	904	895	20	8	5	2	430	420	50	5	8	2	528	478	45
2	9	1	387	406	39	9	0	2	89	133	-89	-4	3	2	1122	1116	19	9	5	2	320	261	-74	6	8	2	100	180	-100
3	9	1	344	116	-86	10	0	2	94	209	-94	-3	3	2	516	558	23	10	5	2	709	599	36	7	8	2	103	60	-103
4	9	1	214	266	-94	11	0	2	95	120	-95	-2	3	2	792	861	27	-10	6	2	195	280	-157	8	8	2	231	158	-137
5	9	1	248	245	-72	-11	1	2	359	235	-62	-1	3	2	1383	1438	72	-9	6	2	535	570	40	-8	9	2	240	196	-114
6	9	1	489	503	40	-10	1	2	278	221	-71	0	3	2	780	821	19	-8	6	2	135	186	-135	-7	9	2	97	156	-97
7	9	1	230	135	-94	-9	1	2	633	658	31	1	3	2	1390	1438	18	-7	6	2	88	199	-88	-6	9	2	228	180	-108
8	9	1	330	369	-71	-8	1	2	672	578	28	2	3	2	828	863	26	-6	6	2	341	395	53	-5	9	2	241	281	-91
-7	10	1	425	371	-75	-7	1	2	1417	1334	22	3	3	2	493	512	29	-5	6	2	859	875	29	-4	9	2	518	542	44
-6	10	1	101	187	-71	-6	1	2	817	791	22	4	3	2	1111	1123	22	-4	6	2	446	554	41	-3	9	2	229	283	-108
-5	10	1	319	344	-67	-5	1	2	1414	1363	19	5	3	2	923	870	22	-3	6	2	1178	1294	30	-2	9	2	597	555	42
-4	10	1	357	265	45	-4	1	2	644	560	18	6	3	2	717	680	28	-2	6	2	649	666	30	-1	9	2	339	293	43
-3	10	1	131	53	-83	-3	1	2	572	491	23	7	3	2	1124	1100	25	-1	6	2	385	421	28	0	9	2	154	120	-81
-2	10	1	116	127	-88	-2	1	2	1018	988	16	8	3	2	520	542	39	0	6	2	370	389	30	1	9	2	320	310	48
-1	10	1	531	442	51	-1	1	2	2939	2659	41	9	3	2	501	490	41	1	6	2	453	445	36	2	9	2	615	601	37
0	10	1	97	69	-48	0	1	2	1976	1884	124	10	3	2	579	571	40	2	6	2	641	670	31	3	9	2	275	261	-91
1	10	1	523	467	23	1	1	2	2801	2695	119	11	3	2	273	273	-98	3	6	2	1235	1312	28	4	9	2	566	526	42
2	10	1	286	145	-73	2	1	2	960	939	18	-10	4	2	305	249	-68	4	6	2	620	587	33	5	9	2	309	278	-80
3	10	1	96	61	-68	3	1	2	462	481	29	-9	4	2	610	658	33	5	6	2	944	879	30	6	9	2	103	192	-103
4	10	1	320	250	47	4	1	2	645	642	20	-8	4	2	566	580	31	6	6	2	319	372	-69	7	9	2	104	158	-104
5	10	1	329	336	50	5	1	2	1374	1348	20	-7	4	2	364	390	44	7	6	2	98	200	-98	8	9	2	180	187	-180
6	10	1	274	176	-125	6	1	2	814	763	23	-6	4	2	1006	983	25	8	6	2	183	214	-183	-7	10	2	100	79	-100
7	10	1	374	381	51	7	1	2	1397	1364	23	-5	4	2	1160	1151	23	9	6	2	587	573	43	-6	10	2	146	120	-146
-6	11	1	302	276	-61	8	1	2	599	572	33	-4	4	2	614	639	24	10	6	2	222	236	-162	-5	10	2	265	292	-92
-5	11	1	341	188	-64	9	1	2	628	654	31	-3	4	2	1011	1112	27	-9	7	2	146	235	-146	-4	10	2	217	253	-133
-4	11	1	104	133	-104	10	1	2	235	224	-97	-2	4	2	1075	1122	26	-8	7	2	320	390	-65	-3	10	2	385	412	59
-3	11	1	305	216	-66	11	1	2	157	231	-157	-1	4	2	666	759	39	-7	7	2	148	33	-148	-1	10	2	256	193	-71
-2	11	1	189	166	-92	-11	2	2	329	318	-66	0	4	2	906	972	25	-6	7	2	372	416	51	0	10	2	353	311	46

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.

H	K	L	10FO	10FC	10S
1	10	2	279	210	-63
2	10	2	356	301	-62
3	10	2	457	405	50
4	10	2	228	219	-127
5	10	2	348	278	-75
6	10	2	285	131	-94
7	10	2	108	73	-108
-6	11	2	107	89	-107
-5	11	2	103	133	-103
-4	11	2	298	316	-86
-3	11	2	215	217	-140
-2	11	2	251	216	-109
-1	11	2	392	315	43
0	11	2	99	105	-70
1	11	2	407	324	-72
2	11	2	214	211	-152
3	11	2	378	193	-64
4	11	2	372	291	-65
5	11	2	107	124	-107
6	11	2	216	90	-178
-4	12	2	274	89	-100
-3	12	2	276	171	-101
-2	12	2	307	205	-58
-1	12	2	121	153	-90
0	12	2	421	264	42
1	12	2	152	147	-86
2	12	2	375	201	-72
3	12	2	106	162	-106
4	12	2	106	87	-106
0	13	2	183	133	-93
1	0	3	639	600	9
2	0	3	1749	1714	19
3	0	3	1851	1751	20
4	0	3	1473	1407	20
5	0	3	885	815	20
6	0	3	951	914	20
7	0	3	172	113	-122
8	0	3	550	530	33
9	0	3	652	601	30
10	0	3	800	813	29
-11	1	3	522	603	42
-10	1	3	659	652	31
-9	1	3	821	805	26
-8	1	3	534	513	33
-7	1	3	337	239	45
-6	1	3	467	461	28
-5	1	3	1128	1059	19
-4	1	3	1715	1682	24
-3	1	3	2158	1998	30
-2	1	3	1770	1690	18
-1	1	3	401	497	20
0	1	3	339	318	15
1	1	3	386	275	15
2	1	3	1771	1741	23
3	1	3	2094	2026	23
4	1	3	1689	1705	21
5	1	3	1093	1086	21
6	1	3	448	456	34
7	1	3	262	205	-65
8	1	3	489	500	37
9	1	3	824	790	28
10	1	3	671	655	33
11	1	3	568	591	41
-11	2	3	411	485	54
-10	2	3	722	719	30
-9	2	3	631	639	31
-8	2	3	619	595	28
-7	2	3	564	519	28
-6	2	3	703	696	23
-5	2	3	830	717	19
-4	2	3	1613	1519	20
-3	2	3	1732	1703	23
-2	2	3	1392	1420	26
-1	2	3	1160	1149	60
0	2	3	516	527	17
1	2	3	1137	1188	15
2	2	3	1353	1401	24
3	2	3	1619	1680	22
4	2	3	1588	1520	23
5	2	3	755	753	24
6	2	3	675	657	28
7	2	3	526	523	35
8	2	3	562	582	35
9	2	3	650	629	33
10	2	3	731	701	31
11	2	3	518	494	45
-10	3	3	532	523	38
-9	3	3	655	645	30
-8	3	3	646	625	29
-7	3	3	741	720	26
-6	3	3	613	606	26
-5	3	3	927	865	21
-4	3	3	1136	1149	20
-3	3	3	1568	1569	23
-2	3	3	1119	1124	24
-1	3	3	1077	1070	55
0	3	3	1254	1347	20
1	3	3	1044	1063	19
2	3	3	1065	1134	25
3	3	3	1515	1554	26
4	3	3	1087	1114	24
5	3	3	916	931	24
6	3	3	591	607	32
7	3	3	727	692	29
8	3	3	622	598	35
9	3	3	693	662	32
10	3	3	509	518	46
11	3	3	375	415	-67
-10	4	3	474	477	31
-9	4	3	521	556	38
-8	4	3	504	558	37
-7	4	3	692	694	28
-6	4	3	698	708	26
-5	4	3	770	770	24
-4	4	3	933	927	23
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Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.   PAGE 4

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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-3	3	8	1008	998	29	-5	6	8	395	384	56	3	9	8	592	559	40	-6	2	9	550	556	32	-7	5	9	718	702	33
-2	3	8	874	831	29	-4	6	8	837	880	38	4	9	8	218	199	-161	-5	2	9	656	717	27	-6	5	9	416	456	52
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0	3	8	1652	1650	67	-2	6	8	976	947	33	6	9	8	110	48	-110	-3	2	9	1268	1288	27	-4	5	9	360	466	-81
1	3	8	521	538	19	-1	6	8	400	396	34	-5	10	8	114	87	-114	-2	2	9	714	745	26	-3	5	9	332	310	-80
2	3	8	857	828	23	0	6	8	113	178	-81	-4	10	8	424	387	70	-1	2	9	754	744	35	-2	5	9	701	591	39
3	3	8	960	1023	24	1	6	8	398	423	35	-2	10	8	449	363	64	0	2	9	832	796	28	-1	5	9	912	873	19
4	3	8	258	356	-87	2	6	8	837	938	28	-1	10	8	303	160	-64	1	2	9	714	717	16	0	5	9	493	467	26
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9	3	8	307	416	-88	7	6	8	327	302	-82	4	10	8	298	376	-93	6	2	9	537	542	45	5	5	9	558	607	46
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-8	4	8	286	255	-71	-8	7	8	102	118	-102	-2	11	8	213	197	-213	9	2	9	560	630	47	8	5	9	415	448	-70
-7	4	8	612	587	31	-7	7	8	101	36	-101	-1	11	8	141	168	-63	-9	3	9	468	482	46	9	5	9	378	386	-81
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-5	4	8	643	606	32	-5	7	8	579	557	44	1	11	8	157	167	-70	-7	3	9	534	495	34	-7	6	9	410	429	57
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-3	4	8	495	460	45	-3	7	8	956	923	37	3	11	8	108	286	-108	-5	3	9	596	616	34	-5	6	9	304	228	-86
-2	4	8	973	956	30	-2	7	8	108	238	-108	4	11	8	110	102	-110	-4	3	9	1052	965	29	-4	6	9	107	244	-107
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0	4	8	539	547	21	0	7	8	94	71	-67	2	0	9	883	899	19	-2	3	9	910	1000	27	-2	6	9	690	652	43
1	4	8	1063	1038	28	1	7	8	428	346	-99	3	0	9	1544	1582	22	-1	3	9	790	802	51	-1	6	9	397	397	-78

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.   PAGE 8

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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-6	7	9	318	369	-85	1	11	9	350	204	-79	-4	3	10	550	544	52	-1	6	10	510	453	30	3	10	10	105	343	-105
-5	7	9	421	433	64	2	11	9	218	199	-166	-3	3	10	745	678	37	0	6	10	437	419	33	4	10	10	342	213	-80
-3	7	9	109	169	-109	3	11	9	172	74	-172	-2	3	10	731	707	34	1	6	10	468	469	69	-1	11	10	127	182	-93
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-5	8	9	108	209	-108	-6	1	10	908	900	26	-7	4	10	451	465	46	-1	7	10	345	365	45	-7	1	11	181	155	-181
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-3	8	9	177	98	-177	-4	1	10	318	350	-56	-5	4	10	664	648	36	1	7	10	329	344	-69	-5	1	11	815	799	30
-2	8	9	451	354	63	-3	1	10	368	302	35	-4	4	10	569	545	53	2	7	10	464	474	45	-4	1	11	649	641	27
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7	8	9	366	296	-83	6	1	10	918	903	30	5	4	10	600	631	45	-3	8	10	399	522	-81	5	1	11	709	754	40
-6	9	9	169	210	-169	7	1	10	887	921	31	6	4	10	660	636	42	-2	8	10	525	408	58	6	1	11	111	249	-111
-5	9	9	327	261	-91	8	1	10	590	641	43	7	4	10	412	462	-70	-1	8	10	300	288	-54	7	1	11	106	137	-106
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-2	9	9	112	250	-112	-8	2	10	670	620	31	-7	5	10	309	343	-74	2	8	10	401	377	54	-7	2	11	277	234	-70
-1	9	9	500	428	35	-7	2	10	879	834	25	-6	5	10	387	419	61	3	8	10	455	536	54	-6	2	11	427	409	42
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1	9	9	447	420	52	-5	2	10	514	496	33	-4	5	10	698	753	46	5	8	10	327	342	-90	-4	2	11	1055	1069	32
2	9	9	292	225	-74	-4	2	10	443	431	66	-3	5	10	838	753	40	6	8	10	112	205	-112	-3	2	11	654	633	32
3	9	9	100	81	-100	-3	2	10	661	654	32	-2	5	10	625	635	45	-5	9	10	330	253	-100	-2	2	11	972	991	24
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-2	10	9	284	179	-119	3	2	10	697	694	27	4	5	10	728	717	35	1	9	10	173	234	-98	4	2	11	987	1016	27

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(exo-2-norbornyl)platinum(II), 1.

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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6	2	11	254	406	-154	8	5	11	333	451	-108	0	10	11	183	108	-89	1	3	12	985	991	21	-3	7	12	195	182	-195
7	2	11	330	265	-90	-7	6	11	526	578	53	1	10	11	319	309	-66	2	3	12	671	668	27	-2	7	12	741	576	45
8	2	11	532	481	52	-6	6	11	492	345	56	2	10	11	102	126	-102	3	3	12	407	383	43	-1	7	12	214	142	-101
9	2	11	185	338	-185	-5	6	11	342	397	-91	3	10	11	277	177	-112	4	3	12	681	654	33	0	7	12	133	62	-96
-9	3	11	391	477	61	-4	6	11	227	228	-227	0	0	12	1543	1553	24	5	3	12	598	593	45	1	7	12	111	136	-85
-8	3	11	422	390	53	-3	6	11	226	240	-226	1	0	12	396	381	25	6	3	12	479	456	60	2	7	12	621	594	36
-7	3	11	396	326	50	-2	6	11	481	443	64	2	0	12	1017	1015	21	7	3	12	697	719	50	3	7	12	303	172	-77
-6	3	11	446	489	45	-1	6	11	659	622	24	3	0	12	225	100	-71	8	3	12	126	207	-126	4	7	12	652	660	42
-5	3	11	558	568	44	0	6	11	582	497	26	4	0	12	419	400	40	-8	4	12	320	466	-78	5	7	12	111	228	-111
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-1	3	11	537	539	28	4	6	11	101	247	-101	8	0	12	733	732	37	-4	4	12	305	290	-129	-2	8	12	124	141	-124
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1	3	11	543	533	21	6	6	11	387	346	-77	-7	1	12	1052	1049	27	-2	4	12	587	579	45	0	8	12	178	88	-79
2	3	11	592	582	27	7	6	11	605	573	52	-6	1	12	367	382	59	-1	4	12	472	410	37	1	8	12	284	304	-83
3	3	11	924	955	25	-7	7	11	284	256	-116	-5	1	12	805	722	38	0	4	12	840	865	20	2	8	12	264	147	-84
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5	3	11	597	583	44	-5	7	11	269	243	-128	-3	1	12	84	252	-84	2	4	12	523	569	35	4	8	12	279	192	-74
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-8	4	11	359	458	-62	0	7	11	667	623	46	2	1	12	347	329	40	7	4	12	247	85	-181	0	9	12	106	17	-75
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-6	4	11	457	450	51	2	7	11	373	391	54	4	1	12	334	245	-58	-7	5	12	459	391	57	2	9	12	494	425	49
-5	4	11	593	519	44	3	7	11	194	230	-194	5	1	12	655	735	42	-6	5	12	338	363	-92	3	9	12	175	137	-175
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-2	4	11	639	660	42	6	7	11	500	536	59	8	1	12	203	233	-203	-3	5	12	410	404	-82	-1	10	12	142	156	-101
-1	4	11	740	715	26	7	7	11	316	262	-125	-8	2	12	628	694	37	-2	5	12	801	682	39	0	10	12	255	235	-95
0	4	11	396	429	31	-6	8	11	312	209	-112	-7	2	12	270	277	-81	-1	5	12	533	519	27	1	10	12	212	150	-88
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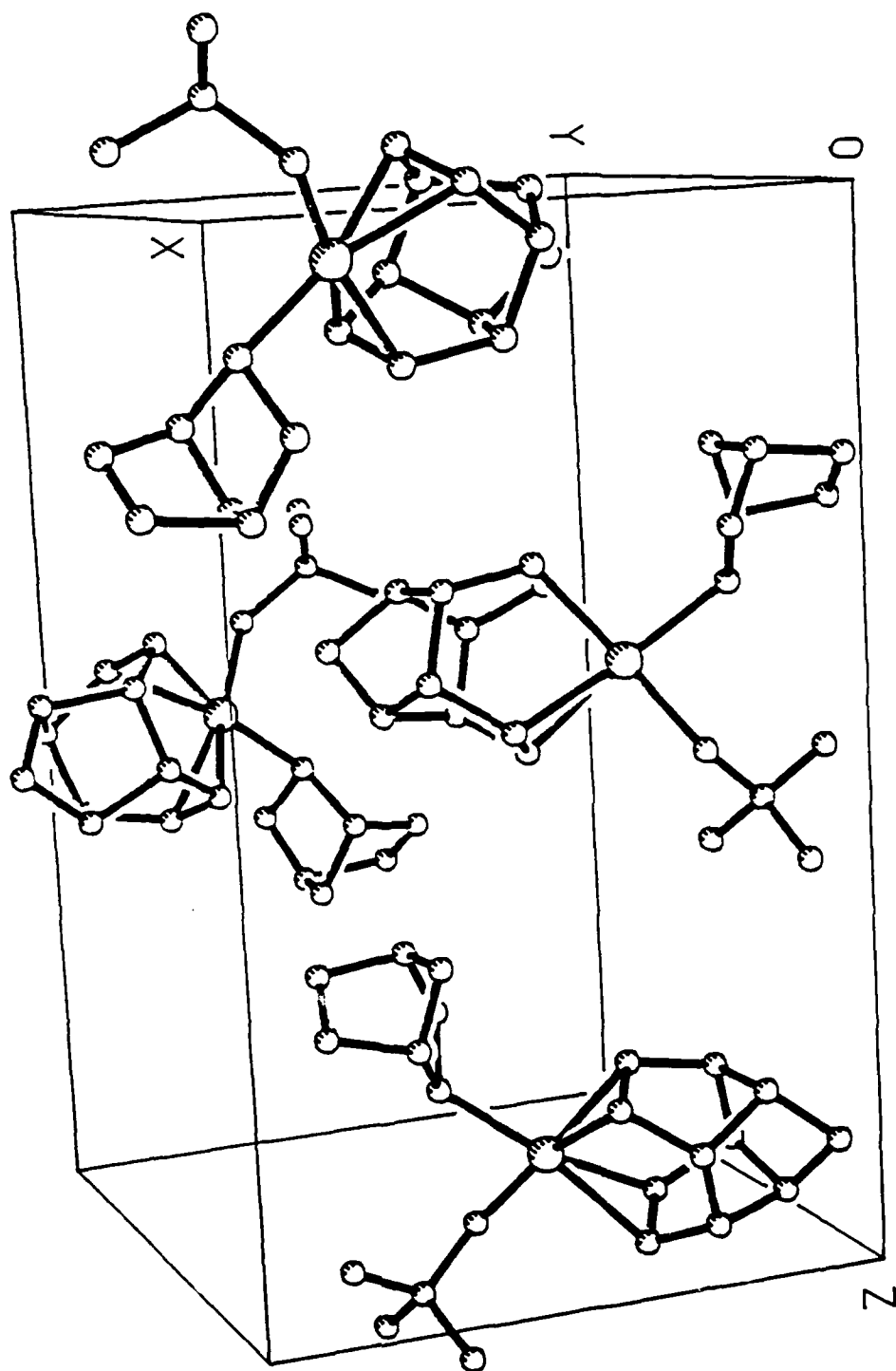
Figure 1a. Packing diagram for **1**.
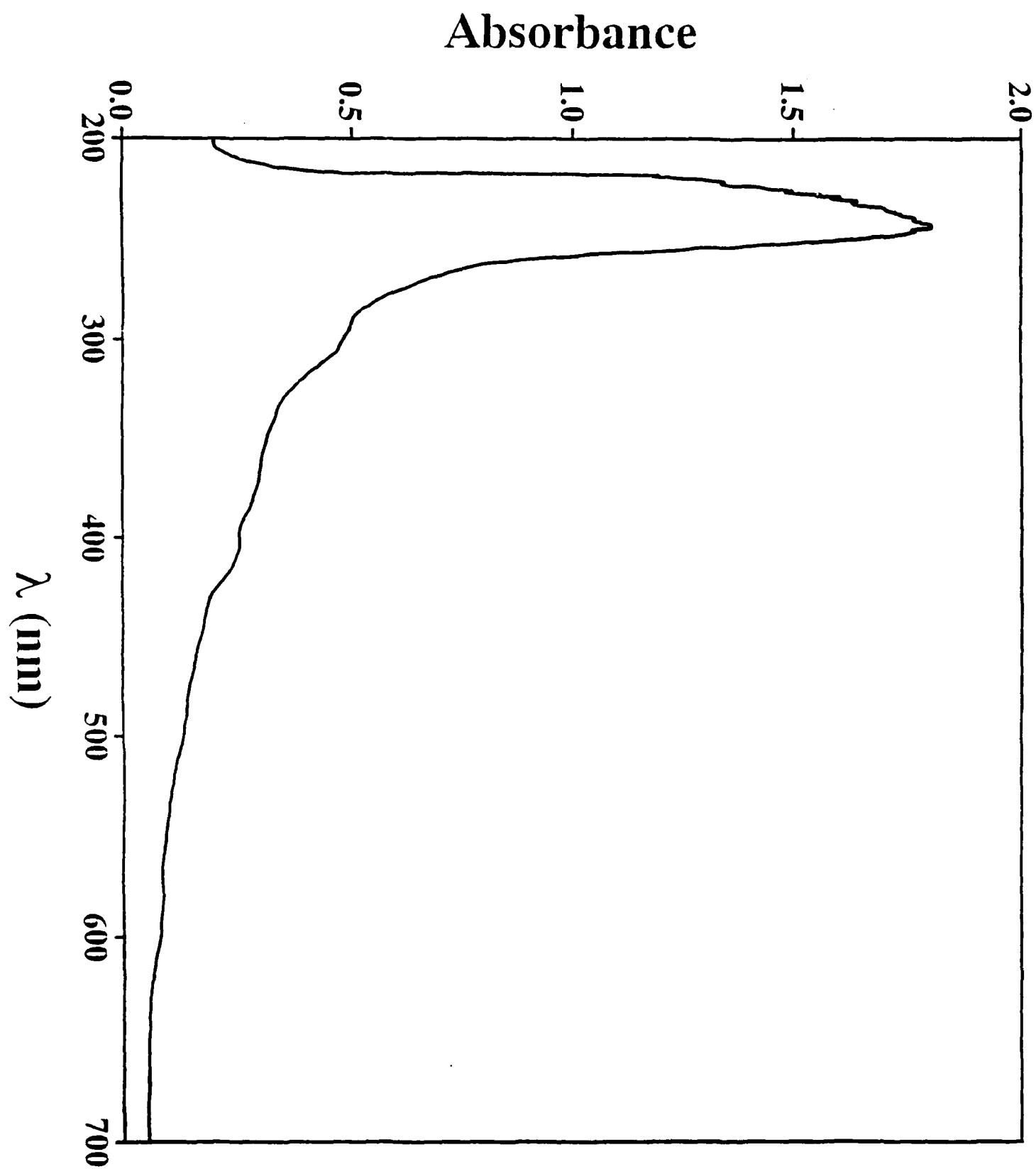
Figure 1b. The UV absorption spectrum of a mixture of 90% **1** and 10% **2** in *n*-hexane.

X-ray Crystallography for (Homohypostrophene)neopentyl-(*endo*norbornyl)platinum(II) 2. We grew x-ray quality crystals of **2** by low temperature recrystallization from diethyl ether/methanol, and mounted them in air as follows. We attached a crystal of dimensions 0.10 x 0.10 x 0.30 mm to a 0.30 mm glass fiber with a minimum amount of silicon grease. We glued the fiber to a 1/8" diameter brass pin using epoxy, and attached the pin to the goniometer head. We transferred the goniometer head to the diffractometer where the crystal was bathed by a cold nitrogen stream (-58 (1) °C).

We used the data from a random search of reciprical space to index the unit cell. A lattice determination using both the P3 program and XCELL suggested a primitive monoclinic cell. Examination of the axial photographs confirmed this assignment. We obtained the final unit cell parameters by performing a least squares refinement of 47 selected reflections, including ten Friedel pairs, in the range $15° < 2\theta < 30°$.

We collected a total of 3641 reflections in the range $4° < 2\theta < 48°$ (*-h, -1, l* to *h, k, l*). Of these, 2888 were unique reflections, and 1951 with $F_O > 6\sigma(F_O)$ were used in the structure solution. We measured the intensities of three check reflections, (-2, 8, 3), (0, 2, 6) and (6, -1, -6), after every 60 reflections. These check reflections showed that the crystal did not decay during the 59 hours of exposure.

Systematic absences uniquely determined the space group to be $P2_1/n$. Successful solution in this space group confirmed its choice. We located the platinum atom from a Patterson synthesis, and used standard difference map techniques to find the remaining non-hydrogen atoms. After all of the non-hydrogen atoms were located and refined anisotropically, a difference map

revealed approximately two-thirds of the hydrogen atom positions. The hydrogen atoms were placed in calculated positions (U_{iso}(H) = 1.2U_{iso}(C); d_{C-H} = 0.96 Å) for refinement. We performed the refinement to convergence (Δ/σ(max) < 0.001) with this model. The weighting scheme was w = [σ^2(F) + gF^2]$^{-1}$ (g= 3.4 x 10^{-4}). The final difference map contained two peaks (1.55 and 1.28 eÅ^{-3}) located near the platinum atom. All other peaks were less than 0.88 eÅ^{-3}.

The molecule crystallizes as a racemate, with one unique crystallographically independent molecule. We attempted a number of semi-empirical absorption corrections. Corrections employing only scans near χ = 270, using either ABSCOR (public domain) or XEMP (Siemens), yielded one non-positive definite thermal ellipse. In the end, we used XEMP to apply an absorption correction which was based on scans from 6 reflections in the range 12° < 2θ < 36° and equivalent data (intensity > 25σ). Refinement yielded a structure with no non-positive definite atoms.

Table IIa. Summary of the crystallographic data for **2**

Color of crystal	yellow
Empirical formula	$C_{23}H_{34}Pt$
Crystal dimensions, mm	0.10 x 0.10 x 0.30
Space group	$P2_1/n$ (No. 8)
Cell dimensions	
a, Å	10.679 (5)
b, Å	17.085 (7)
c, Å	11.514 (4)
β, deg	116.77 (3)
Temperature, °C	-58 (1)
Wavelength, Å	0.71073
Z (molecules/cell)	4
Volume, Å^3	1875.4 (14)
d_{calcd}, g cm^{-3}	1.79
Linear absorption coefficient, cm^{-1}	75.5
Scan type	$\theta - 2\theta$
Scan speed, deg/min	3 - 30
Scan width, deg (+ dispersion)	1.60
Background / scan ratio	0.50
2θ range, deg	4 - 48
Data collected	$h, k, \pm l$
$F(000)$	1000
Parameters refined	217
Total number of reflections collected	3641
Number of unique reflections	2888
R_{int}	0.0370
Number with $F_o > 6.00\sigma(F_o)$	1951
$R(F)$	0.0415
$R_w(F)$	0.0394
"Goodness of fit" for last cycle	1.50
Largest Δ/σ for last cycle	0.001
Final difference map (maximum)	1.55 and 1.28 eÅ^{-3} near Pt
(remainder)	<0.88 eÅ^{-3}

[a] $R = \Sigma |(Fo-Fc)|/\Sigma Fo$

[b] $R_w = \Sigma(w^{1/2}|(Fo-Fc)|/\Sigma(w^{1/2}Fo)$, $w = [\sigma^2(F) + gF^2]^{-1}$

Table IIb. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 2[a]

atom	x	y	z	U(eq)
Pt	6333(1)	2112(1)	1750(1)	20(1)
C(1)	10599(15)	2240(8)	1269(12)	30(6)
C(2)	9440(13)	1632(8)	874(11)	25(5)
C(3)	9933(14)	2770(7)	1853(11)	23(5)
C(4)	9261(14)	1456(8)	2123(11)	23(5)
C(5)	8033(14)	2068(8)	126(11)	25(5)
C(6)	8418(14)	2899(9)	826(10)	26(5)
C(7)	9658(13)	2280(7)	2830(10)	21(5)
C(8)	7779(14)	1209(8)	1580(11)	24(6)
C(9)	7022(14)	1583(8)	375(11)	27(6)
C(10)	7667(13)	3086(7)	1666(10)	17(5)
C(11)	8389(14)	2711(8)	2815(12)	28(6)
C(12)	5133(14)	2860(9)	2296(10)	28(5)
C(13)	3778(15)	3207(8)	1166(12)	27(6)
C(14)	4190(17)	3776(9)	347(11)	39(6)
C(15)	4713(18)	4512(9)	1201(12)	39(7)
C(16)	4649(17)	4244(9)	2485(12)	40(7)
C(17)	5771(15)	3607(9)	3100(11)	33(6)
C(18)	3297(15)	3768(9)	1899(12)	34(6)
C(19)	4678(14)	1316(9)	1224(11)	29(6)
C(20)	4591(15)	685(8)	2167(12)	29(6)
C(21)	5723(15)	64(9)	2546(13)	37(6)
C(22)	3155(16)	277(10)	1490(14)	46(7)
C(23)	4709(19)	1045(9)	3430(12)	45(8)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table IIc. Complete listing of bond lengths (Å) for **2**

Pt-C(8)	2.253 (15)	C(6)-C(10)	1.543 (22)
Pt-C(9)	2.217 (16)	C(7)-C(11)	1.536 (21)
Pt-C(10)	2.220 (13)	C(8)-C(9)	1.405 (16)
Pt-C(11)	2.224 (13)	C(10)-C(11)	1.354 (16)
Pt-C(12)	2.096 (16)	C(12)-C(13)	1.563 (16)
Pt-C(19)	2.092 (15)	C(12)-C(17)	1.544 (19)
C(1)-C(2)	1.520 (20)	C(13)-C(14)	1.550 (23)
C(1)-C(3)	1.486 (22)	C(13)-C(18)	1.512 (23)
C(2)-C(4)	1.563 (21)	C(14)-C(15)	1.539 (19)
C(2)-C(5)	1.546 (18)	C(15)-C(16)	1.579 (23)
C(3)-C(6)	1.530 (16)	C(16)-C(17)	1.535 (21)
C(3)-C(7)	1.532 (20)	C(16)-C(18)	1.524 (22)
C(4)-C(7)	1.585 (17)	C(19)-C(20)	1.563 (21)
C(4)-C(8)	1.478 (19)	C(20)-C(21)	1.516 (21)
C(5)-C(6)	1.593 (19)	C(20)-C(22)	1.539 (20)
C(5)-C(9)	1.488 (23)	C(20)-C(23)	1.531 (21)

Table IId. Complete listing of bond angles (°) for **2**

C(8)-Pt-C(9)	36.6(4)	C(2)-C(5)-C(9)	103.4(11)
C(8)-Pt-C(10)	91.8(5)	C(6)-C(5)-C(9)	116.3(13)
C(9)-Pt-C(10)	81.3(5)	C(3)-C(6)-C(5)	101.8(10)
C(8)-Pt-C(11)	79.5(5)	C(3)-C(6)-C(10)	102.3(9)
C(9)-Pt-C(11)	91.1(5)	C(5)-C(6)-C(10)	114.4(12)
C(10)-Pt-C(11)	35.5(4)	C(3)-C(7)-C(4)	101.8(10)
C(8)-Pt-C(12)	168.2(5)	C(3)-C(7)-C(11)	100.9(10)
C(9)-Pt-C(12)	155.1(4)	C(4)-C(7)-C(11)	112.9(10)
C(10)-Pt-C(12)	92.7(6)	Pt-C(8)-C(4)	114.4(9)
C(11)-Pt-C(12)	98.0(5)	Pt-C(8)-C(9)	70.3(9)
C(8)-Pt-C(19)	93.0(6)	C(4)-C(8)-C(9)	108.6(13)
C(9)-Pt-C(19)	92.1(6)	Pt-C(9)-C(5)	114.4(9)
C(10)-Pt-C(19)	161.7(4)	Pt-C(9)-C(8)	73.1(9)
C(11)-Pt-C(19)	162.6(5)	C(5)-C(9)-C(8)	107.4(11)
C(12)-Pt-C(19)	86.1(6)	Pt-C(10)-C(6)	113.4(8)
C(2)-C(1)-C(3)	93.5(12)	Pt-C(10)-C(11)	72.4(8)
C(1)-C(2)-C(4)	106.0(10)	C(6)-C(10)-C(11)	106.6(12)
C(1)-C(2)-C(5)	107.1(11)	Pt-C(11)-C(7)	115.6(8)
C(4)-C(2)-C(5)	96.5(12)	Pt-C(11)-C(10)	72.1(7)
C(1)-C(3)-C(6)	107.0(10)	C(7)-C(11)-C(10)	110.4(13)
C(1)-C(3)-C(7)	106.8(11)	Pt-C(12)-C(13)	116.3(9)
C(6)-C(3)-C(7)	99.2(11)	Pt-C(12)-C(17)	121.3(11)
C(2)-C(4)-C(7)	100.7(11)	C(13)-C(12)-C(17)	100.3(11)
C(2)-C(4)-C(8)	102.5(9)	C(12)-C(13)-C(14)	109.5(13)
C(7)-C(4)-C(8)	117.3(12)	C(12)-C(13)-C(18)	101.3(10)
C(2)-C(5)-C(6)	100.5(9)	C(14)-C(13)-C(18)	101.7(12)
C(13)-C(14)-C(15)	104.2(12)	Pt-C(19)-C(20)	123.7(7)
C(14)-C(15)-C(16)	101.8(12)	C(19)-C(20)-C(21)	113.2(14)
C(15)-C(16)-C(17)	106.5(14)	C(19)-C(20)-C(22)	108.5(10)
C(15)-C(16)-C(18)	99.9(10)	C(21)-C(20)-C(22)	108.2(12)
C(17)-C(16)-C(18)	102.4(12)	C(19)-C(20)-C(23)	112.2(12)
C(12)-C(17)-C(16)	105.5(10)	C(21)-C(20)-C(23)	107.1(10)
C(13)-C(18)-C(16)	95.3(13)	C(22)-C(20)-C(23)	107.4(15)

Table IIe. Anisotropic displacement parameters ($\mathring{A}^2 \times 10^3$) for **2**[a]

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pt	20(1)	20(1)	19(1)	0(1)	9(1)	-2(1)
C(1)	33(8)	27(8)	37(6)	8(6)	22(6)	8(7)
C(2)	16(7)	31(8)	33(6)	-7(6)	15(6)	-2(6)
C(3)	24(7)	20(8)	28(6)	-3(5)	13(6)	-6(6)
C(4)	24(8)	14(7)	28(6)	6(5)	10(6)	14(6)
C(5)	30(8)	22(7)	23(6)	2(6)	12(6)	-3(7)
C(6)	34(8)	20(7)	29(6)	4(6)	20(6)	-2(8)
C(7)	14(7)	23(8)	21(5)	-2(5)	3(5)	2(6)
C(8)	33(9)	11(7)	34(7)	-11(5)	21(7)	1(6)
C(9)	29(8)	26(8)	27(6)	-9(5)	12(6)	-2(7)
C(10)	13(7)	8(8)	29(6)	-2(5)	8(6)	-2(5)
C(11)	26(8)	33(9)	27(6)	-12(5)	13(6)	-14(6)
C(12)	39(8)	29(8)	22(6)	8(6)	20(6)	-4(8)
C(13)	21(8)	23(7)	36(7)	-12(6)	12(6)	-7(6)
C(14)	54(11)	33(8)	24(6)	0(6)	14(7)	18(9)
C(15)	55(11)	26(8)	39(7)	16(6)	24(8)	10(8)
C(16)	61(12)	29(9)	34(7)	3(6)	25(8)	6(8)
C(17)	36(9)	38(9)	27(6)	-1(6)	15(6)	2(8)
C(18)	31(9)	32(9)	40(7)	1(7)	17(7)	2(8)
C(19)	26(8)	42(9)	27(6)	18(6)	20(6)	18(8)
C(20)	21(8)	26(8)	31(7)	3(6)	4(6)	-6(7)
C(21)	27(9)	35(9)	45(8)	4(7)	13(7)	-6(7)
C(22)	35(10)	50(11)	57(9)	-3(8)	25(8)	-17(9)
C(23)	69(13)	34(9)	38(8)	11(6)	29(8)	4(9)

[a] The anisotropic displacement exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \ldots + 2hka^* b^* U_{12})$.

Table IIf. Coordinates of hydrogen atoms (x 10^4) for **2**

atom	x	y	z
H(1A)	10648	2464	526
H(1B)	11504	2056	1890
H(2A)	9560	1177	445
H(3A)	10440	3246	2203
H(4A)	9876	1046	2637
H(5A)	7786	2103	-784
H(6A)	8332	3321	244
H(7A)	10440	2264	3681
H(8A)	7583	667	1642
H(9A)	6349	1280	-334
H(10A)	7369	3613	1694
H(11A)	8512	2993	3581
H(12A)	4833	2541	2811
H(13A)	3088	2820	677
H(14A)	3399	3911	-459
H(14B)	4908	3549	169
H(15A)	4152	4967	811
H(15B)	5669	4623	1397
H(16A)	4672	4661	3052
H(17A)	6630	3747	3075
H(17B)	5963	3521	3989
H(18A)	3122	3517	2558
H(18B)	2490	4067	1334
H(19A)	4624	1031	484
H(19B)	3829	1616	931
H(21A)	6642	290	2984
H(21B)	5592	-303	3114
H(21C)	5628	-202	1776
H(22A)	3074	-111	2054
H(22B)	2425	659	1275
H(22C)	3070	32	708
H(23A)	5595	1305	3892
H(23B)	3963	1419	3191
H(23C)	4610	650	3977

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(endo-2-norbornyl)platinum(II), 2.   PAGE 1

H	K	L	10FO	10FC	10S
2	0	0	873	899	14
4	0	0	1687	1724	19
6	0	0	764	692	23
8	0	0	1033	1052	29
10	0	0	743	-664	39
1	1	0	269	-224	11
2	1	0	1522	1544	12
3	1	0	584	604	14
4	1	0	376	370	20
5	1	0	235	99	37
6	1	0	2021	1993	17
7	1	0	347	-404	40
8	1	0	447	-379	39
9	1	0	192	-63	-103
10	1	0	867	-824	24
1	2	0	1495	1546	13
3	2	0	612	671	19
4	2	0	1981	1964	21
5	2	0	805	-864	19
6	2	0	726	-677	23
7	2	0	439	-323	44
8	2	0	1386	-1262	27
9	2	0	522	417	49
10	2	0	566	493	47
1	3	0	412	407	12
2	3	0	2209	2259	24
3	3	0	1201	-1196	17
4	3	0	325	260	31
5	3	0	292	-426	45
6	3	0	1004	-974	21
7	3	0	1292	1272	25
9	3	0	399	387	64
10	3	0	691	718	44
2	4	0	70	-112	-140
3	4	0	1216	-1252	17
4	4	0	1225	-1187	19
5	4	0	1468	1423	20
6	4	0	615	564	27
7	4	0	389	468	57
8	4	0	813	783	30
9	4	0	822	-842	33
10	4	0	280	-282	-96
3	5	0	2292	2359	21
5	5	0	857	858	21
6	5	0	736	704	24
7	5	0	1372	-1277	25
8	5	0	106	-48	-213
9	5	0	282	-338	-85
10	5	0	317	-279	-75
0	6	0	77	-117	-155
1	6	0	1261	1242	21
2	6	0	295	-411	36
3	6	0	1345	1405	19
4	6	0	141	-95	-83
5	6	0	1557	-1522	21
6	6	0	309	-275	-53
7	6	0	682	-626	33
8	6	0	212	-234	-111
9	6	0	1193	1156	28
10	6	0	110	95	-220
1	7	0	1559	1506	16
2	7	0	252	-274	-47
3	7	0	1993	-1950	20
4	7	0	200	93	-62
5	7	0	1105	-1084	21
6	7	0	93	119	-184
7	7	0	1397	1338	27
8	7	0	377	-158	63
9	7	0	256	313	-96
10	7	0	242	-88	-101
0	8	0	478	-498	30
1	8	0	1346	-1365	30
2	8	0	395	258	32
3	8	0	816	-841	19
4	8	0	512	551	29
5	8	0	1440	1459	23
6	8	0	269	-269	-69
7	8	0	674	635	35
8	8	0	534	-545	50
9	8	0	899	-916	32
1	9	0	1274	-1274	16
3	9	0	1051	1143	19
4	9	0	192	-222	-79
5	9	0	829	835	25
6	9	0	870	-836	25
7	9	0	1155	-1161	27
8	9	0	434	382	53
9	9	0	193	-217	-118
0	10	0	1569	1545	19
1	10	0	957	916	21
2	10	0	286	-385	-53
3	10	0	485	489	32
4	10	0	1235	-1239	21
5	10	0	849	-805	25
6	10	0	447	412	45
7	10	0	526	-479	45
8	10	0	905	865	31
9	10	0	575	577	45
1	11	0	349	283	40
2	11	0	1488	-1440	21
3	11	0	741	-738	24
4	11	0	214	-99	-79
5	11	0	369	-323	51
6	11	0	1226	1212	23
7	11	0	709	753	36
8	11	0	488	-533	51
9	11	0	111	211	-222
0	12	0	2149	-2072	24
1	12	0	313	-273	47
2	12	0	227	256	-83
3	12	0	152	-83	-114
4	12	0	1348	1330	22
5	12	0	232	144	-81
6	12	0	493	-531	43
7	12	0	107	129	-214
8	12	0	1012	-936	28
1	13	0	234	38	-82
3	13	0	95	155	-188
4	13	0	248	-201	-72
5	13	0	97	-55	-193
6	13	0	1235	-1271	25
7	13	0	206	-53	-109
8	13	0	321	375	-75
1	14	0	380	-351	52
2	14	0	306	64	-65
3	14	0	94	-191	-188
4	14	0	1209	-1186	21
5	14	0	373	462	56
6	14	0	275	352	-82
7	14	0	116	89	-234
1	15	0	490	-466	33
2	15	0	1308	-1243	21
3	15	0	425	365	44
4	15	0	235	182	-87
5	15	0	287	333	-75
6	15	0	871	844	33
7	15	0	549	-435	51
0	16	0	958	-975	25
1	16	0	655	602	25
2	16	0	216	-27	-93
3	16	0	461	459	47
4	16	0	773	775	31
5	16	0	692	-686	33
6	16	0	321	-359	-80
1	17	0	658	657	24
2	17	0	684	657	33
3	17	0	893	-880	27
4	17	0	106	-143	-213
5	17	0	321	-368	-77
0	18	0	532	508	42
1	18	0	756	-737	22
2	18	0	160	126	-124
3	18	0	554	-526	43
1	19	0	701	-699	26
2	19	0	232	-26	-97
-11	0	1	437	352	60
-9	0	1	1382	-1290	27
-7	0	1	102	-36	-205
-3	0	1	146	-214	-93
-1	0	1	3276	-3294	10
1	0	1	1258	1273	9
3	0	1	2226	2185	25
5	0	1	1020	-957	19
7	0	1	1519	-1490	25
9	0	1	735	739	35
-11	1	1	900	-872	24
-10	1	1	115	133	-163
-9	1	1	345	-97	57
-8	1	1	373	175	50
-7	1	1	1796	1705	19
-6	1	1	554	-497	23
-5	1	1	296	-177	37
-4	1	1	377	-342	26
-2	1	1	194	227	22
0	1	1	460	428	7
1	1	1	3036	2937	8
2	1	1	890	-852	12
3	1	1	1533	-1455	14
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-5	3	4	1452	1508	18	-6	6	4	304	-226	50	-6	9	4	230	-206	-80	0	12	4	646	617	18	1	16	4	533	-514	24
-2	3	4	469	484	16	-5	6	4	166	103	-80	-4	9	4	717	766	25	1	12	4	394	420	30	2	16	4	761	-730	28
-1	3	4	1918	-1955	13	-4	6	4	182	-178	-67	-3	9	4	465	523	31	2	12	4	1337	-1279	21	3	16	4	457	501	51
0	3	4	1567	-1523	13	-3	6	4	2359	2226	23	-1	9	4	1869	1878	15	3	12	4	190	-38	-86	-5	17	4	674	721	39
1	3	4	569	547	22	-2	6	4	195	208	-52	0	9	4	1122	-1107	12	4	12	4	102	-31	-206	-3	17	4	329	-359	-65
2	3	4	652	-649	21	-1	6	4	1169	-1171	13	1	9	4	701	-706	17	5	12	4	104	-188	-208	-2	17	4	321	151	-61
3	3	4	944	926	19	0	6	4	149	-67	-40	2	9	4	276	-243	-53	6	12	4	1077	969	28	-1	17	4	1019	-946	17
4	3	4	1223	1221	21	1	6	4	1743	-1724	14	3	9		10	-885	23	-9	13	4	165	31	-134	0	17	4	452	-417	31
5	3	4	849	-837	22	2	6	4	351	-368	34	4	9	4	820	783	24	-8	13	4	876	-907	30	1	17	4	194	278	-76
6	3	4	91	-29	-182	3	6	4	809	775	21	5	9	4	845	840	27	-7	13	4	103	-56	-206	2	17	4	212	-225	-100
7	3	4	485	-473	42	4	6	4	86	11	-171	6	9	4	102	-87	-205	-6	13	4	712	-735	31	-4	18	4	254	61	-91
8	3	4	889	-892	26	5	6	4	869	866	24	7	9	4	479	582	56	-5	13	4	98	7	-198	-3	18	4	1022	-1042	27
-11	4	4	908	-937	34	6	6	4	241	5	-73	-10	10	4	429	-366	57	-4	13	4	1084	1116	23	-2	18	4	455	-486	52
-10	4	4	464	-454	56	7	6	4	761	-797	31	-9	10	4	107	-101	-215	-3	13	4	133	111	-132	-1	18	4	237	386	-72
-9	4	4	110	64	-220	8	6	4	101	-39	-202	-8	10	4	769	-734	31	-2	13	4	614	632	31	0	18	4	308	-99	-66
-8	4	4	544	-505	43	-11	7	4	203	143	-130	-7	10	4	975	-989	26	-1	13	4	150	-101	-93	1	18	4	673	618	24
-7	4	4	1453	1429	22	-10	7	4	110	-189	-222	-6	10	4	819	874	27	0	13	4	1404	-1465	14	-11	0	5	1012	988	31
-4	4	4	831	853	22	-9	7	4	1316	-1346	27	-5	10	4	142	184	-125	1	13	4	90	-6	-127	-9	0	5	930	967	31
-5	4	4	177	-197	-70	-8	7	4	107	172	-214	-4	10	4	675	677	27	2	13	4	491	-483	37	-7	0	5	2135	-2136	25
-4	4	4	1133	1064	17	-7	7	4	343	224	54	-3	10	4	669	624	25	3	13	4	100	102	-202	-5	0	5	528	-507	29
-3	4	4	1812	-1740	17	-6	7	4	185	120	-87	-2	10	4	1271	-1238	19	4	13	4	1059	1060	27	-3	0	5	2137	2054	19

Observed and Calculated Structure Factors for (Homohypostrophene)neopentyl(endo-2-norbornyl)platinum(II), 2.

H	K	L	10FO	10FC	10S
-1	0	5	239	-44	29
1	0	5	1867	-1904	15
3	0	5	483	493	26
5	0	5	1265	1282	20
7	0	5	429	-262	42
-12	1	5	120	-37	-169
-11	1	5	461	407	45
-10	1	5	325	-324	-60
-9	1	5	1087	-1045	21
-8	1	5	146	46	-121
-7	1	5	617	-626	24
-6	1	5	511	534	23
-5	1	5	1990	1941	16
-4	1	5	393	-333	21
-3	1	5	638	609	11
-2	1	5	258	-157	19
-1	1	5	2296	-2284	13
0	1	5	526	488	8
1	1	5	298	123	26
2	1	5	233	166	35
3	1	5	1655	1668	14
4	1	5	336	-340	30
5	1	5	312	-214	37
6	1	5	240	-236	-61
7	1	5	1077	-1172	17
8	1	5	294	244	-51
-12	2	5	379	-451	-74
-11	2	5	853	-846	35
-10	2	5	113	103	-226
-9	2	5	639	-673	41
-8	2	5	786	759	31
-7	2	5	1672	1641	23
-6	2	5	270	-229	-54
-5	2	5	457	485	29
-4	2	5	750	-684	19
-3	2	5	1752	-1738	16
-2	2	5	956	933	14
-1	2	5	277	103	33
0	2	5	814	827	12
1	2	5	1925	1896	16
2	2	5	651	-623	19
3	2	5	383	-327	33
4	2	5	447	-394	31
5	2	5	1164	1203	21
6	2	5	502	507	35
7	2	5	301	281	-63
-12	3	5	315	18	-85
-11	3	5	405	-488	67
-10	3	5	709	662	38
-9	3	5	807	804	33
-8	3	5	294	-313	-77
-7	3	5	278	353	-65
-6	3	5	1018	-1095	20
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-1	3	5	1550	1564	14
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1	3	5	151	-148	-56
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3	3	5	1444	-1397	21
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6	3	5	431	412	43
7	3	5	719	749	31
-11	4	5	598	665	47
-10	4	5	254	-298	-95
-9	4	5	412	467	59
-8	4	5	1239	-1244	26
-7	4	5	858	-879	25
-6	4	5	418	363	36
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-3	4	5	1090	1036	17
-2	4	5	1234	-1268	15
-1	4	5	422	-297	21
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3	4	5	230	190	-56
4	4	5	920	888	22
5	4	5	867	866	23
6	4	5	819	-833	28
7	4	5	192	-187	-107
-11	5	5	114	205	-229
-10	5	5	1284	-1298	26
-9	5	5	437	-411	54
-8	5	5	411	467	57
-7	5	5	95	-139	-188
-6	5	5	1468	1468	19
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-3	5	5	526	455	20
-2	5	5	1916	-1921	19
-1	5	5	452	-442	19
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3	5	5	781	770	23
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5	5	5	204	-158	-79
6	5	5	306	-306	-65
7	5	5	309	-334	-76
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-9	6	5	109	1	-218
-8	6	5	1042	1072	29
-7	6	5	95	106	-190
-6	6	5	696	-745	25
-5	6	5	83	-136	-166
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3	6	5	83	29	-166
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-11	7	5	253	37	-100
-10	7	5	1246	1263	26
-9	7	5	234	-151	-98
-8	7	5	499	-514	47
-7	7	5	95	-33	-190
-6	7	5	1443	1436	21
-5	7	5	387	413	40
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-3	7	5	224	99	-50
-2	7	5	1857	1882	18
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1	7	5	201	135	-56
2	7	5	1082	1037	21
3	7	5	191	219	-77
4	7	5	1224	1165	21
5	7	5	93	-88	-185
6	7	5	154	267	-133
7	7	5	327	-99	-72
-11	8	5	375	-235	-67
-10	8	5	240	-300	-102
-9	8	5	416	-362	55
-8	8	5	1171	-1097	25
-7	8	5	232	303	-83
-6	8	5	838	815	23
-5	8	5	214	15	-72
-4	8	5	1281	1236	20
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-2	8	5	1181	-1113	17
-1	8	5	203	4	-39
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3	8	5	86	94	-172
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-9	9	5	650	612	36
-8	9	5	361	333	-63
-7	9	5	166	248	-115
-6	9	5	1086	1046	23
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-2	9	5	1398	-1359	18
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2	9	5	487	423	33
3	9	5	873	-872	23
4	9	5	1062	-1058	22
5	9	5	102	163	-204
6	9	5	448	-410	51
-10	10	5	349	261	-67
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-8	10	5	809	785	31
-7	10	5	1070	-1035	24
-6	10	5	451	-488	43
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2	10	5	908	-860	22
3	10	5	246	202	-64
4	10	5	519	-567	42
5	10	5	871	828	29
6	10	5	570	532	43
-10	11	5	333	406	-75
-9	11	5	801	-823	31
-8	11	5	103	-34	-207
-7	11	5	556	-564	37
-6	11	5	470	-366	41
-5	11	5	937	956	24
-4	11	5	648	675	29
-3	11	5	298	314	-55
-2	11	5	654	618	25
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0	11	5	458	-459	22
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2	11	5	350	-294	48
3	11	5	1234	1202	21
4	11	5	349	350	-60
5	11	5	329	-210	-65
-9	12	5	309	-398	-74
-8	12	5	106	-259	-212
-7	12	5	1324	1421	24
-6	12	5	259	260	-73
-5	12	5	264	238	-69
-4	12	5	367	298	49
-3	12	5	1479	-1486	22
-2	12	5	90	-130	-179
-1	12	5	225	-190	-50
0	12	5	165	-245	-68
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3	12	5	208	-293	-97
4	12	5	160	180	-132
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-8	13	5	106	-45	-212
-7	13	5	445	435	50
-6	13	5	100	-144	-200
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3	13	5	1179	-1196	25
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-6	14	5	211	238	-97
-5	14	5	373	-356	58
-4	14	5	323	284	-57
-3	14	5	1345	1281	21
-2	14	5	287	-264	-62
-1	14	5	97	-22	-138
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1	14	5	1197	-1157	14
2	14	5	222	239	-78
3	14	5	257	138	-83
4	14	5	246	66	-93
-7	15	5	110	-249	-220
-6	15	5	460	463	47
-5	15	5	1075	986	24
-4	15	5	102	-95	-204
-3	15	5	304	303	-64
-2	15	5	385	-296	50
-1	15	5	1008	-936	16
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2	15	5	471	430	39
3	15	5	952	972	29
-6	16	5	108	-248	-217
-5	16	5	327	291	-66
-4	16	5	604	-627	38
-3	16	5	896	-880	18
-2	16	5	592	528	34
-1	16	5	126	185	-112
0	16	5	576	536	22
1	16	5	645	649	24
2	16	5	426	-455	52
-5	17	5	485	-481	50
-4	17	5	518	497	47
-3	17	5	167	-14	-133
-2	17	5	652	680	37
-1	17	5	630	629	26
0	17	5	658	-687	24
1	17	5	197	42	-69
-3	18	5	364	277	-64
-2	18	5	556	-584	43
-1	18	5	300	-150	-67
-12	0	6	766	-770	41

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
-10	0	6	303	184	-85	-5	3	6	834	-797	19	-2	6	6	66	-98	-132	2	9	6	920	854	21	-1	13	6	88	-115	-125
-8	0	6	1784	1807	24	-4	3	6	282	187	37	-1	6	6	606	-564	14	3	9	6	952	928	22	0	13	6	391	444	28
-6	0	6	179	-12	-91	-3	3	6	261	-312	30	0	6	6	201	-183	-41	4	9	6	317	-272	-65	1	13	6	157	-17	-78
-4	0	6	2159	-2128	23	-2	3	6	1633	-1671	17	1	6	6	1774	1750	16	5	9	6	104	136	-209	2	13	6	1116	1040	21
-2	0	6	184	-91	-43	-1	3	6	1479	1463	14	2	6	6	174	-38	-80	-10	10	6	109	-284	-218	3	13	6	102	-78	-204
0	0	6	1933	1944	14	0	3	6	325	326	21	3	6	6	709	696	26	-9	10	6	475	-595	51	-8	14	6	958	1020	28
2	0	6	967	-976	18	1	3	6	440	444	21	4	6	6	89	208	-177	-8	10	6	854	868	28	-7	14	6	191	-81	-120
4	0	6	970	-995	20	2	3	6	1211	1154	21	5	6	6	1266	-1266	23	-7	10	6	544	537	37	-6	14	6	99	-46	-199
6	0	6	493	646	42	3	3	6	924	-904	21	6	6	6	102	-37	-205	-6	10	6	95	-142	-190	-5	14	6	224	-154	-89
-12	1	6	432	-435	47	4	3	6	519	-516	31	-11	7	6	918	-950	32	-5	10	6	643	601	29	-4	14	6	1324	-1305	22
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-9	1	6	111	-16	-158	7	3	6	536	559	45	-8	7	6	155	130	-138	-2	10	6	79	158	-158	-1	14	6	335	320	34
-8	1	6	370	340	44	-11	4	6	521	510	49	-7	7	6	973	938	23	-1	10	6	730	-761	16	0	14	6	959	924	15
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-3	1	6	213	103	28	-6	4	6	86	2	-172	-2	7	6	315	372	32	4	10	6	793	-730	28	-6	15	6	873	-896	30
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2	1	6	1588	-1588	14	0	4	6	790	804	12	3	7	6	1042	-967	21	-7	11	6	441	407	45	-1	15	6	331	-333	36
3	1	6	313	282	31	1	4	6	1359	-1317	14	4	7	6	91	21	-182	-6	11	6	1518	-1529	23	0	15	6	409	-374	41
4	1	6	590	521	20	2	4	6	598	-503	23	5	7	6	99	-66	-198	-5	11	6	594	-540	31	1	15	6	197	-93	-66
5	1	6	158	-12	-85	3	4	6	345	-299	41	6	7	6	248	103	-87	-4	11	6	93	77	-184	2	15	6	973	-868	25
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7	1	6	364	-284	57	5	4	6	1130	1144	23	-10	8	6	108	201	-217	-2	11	6	1078	1094	20	-5	16	6	453	489	51
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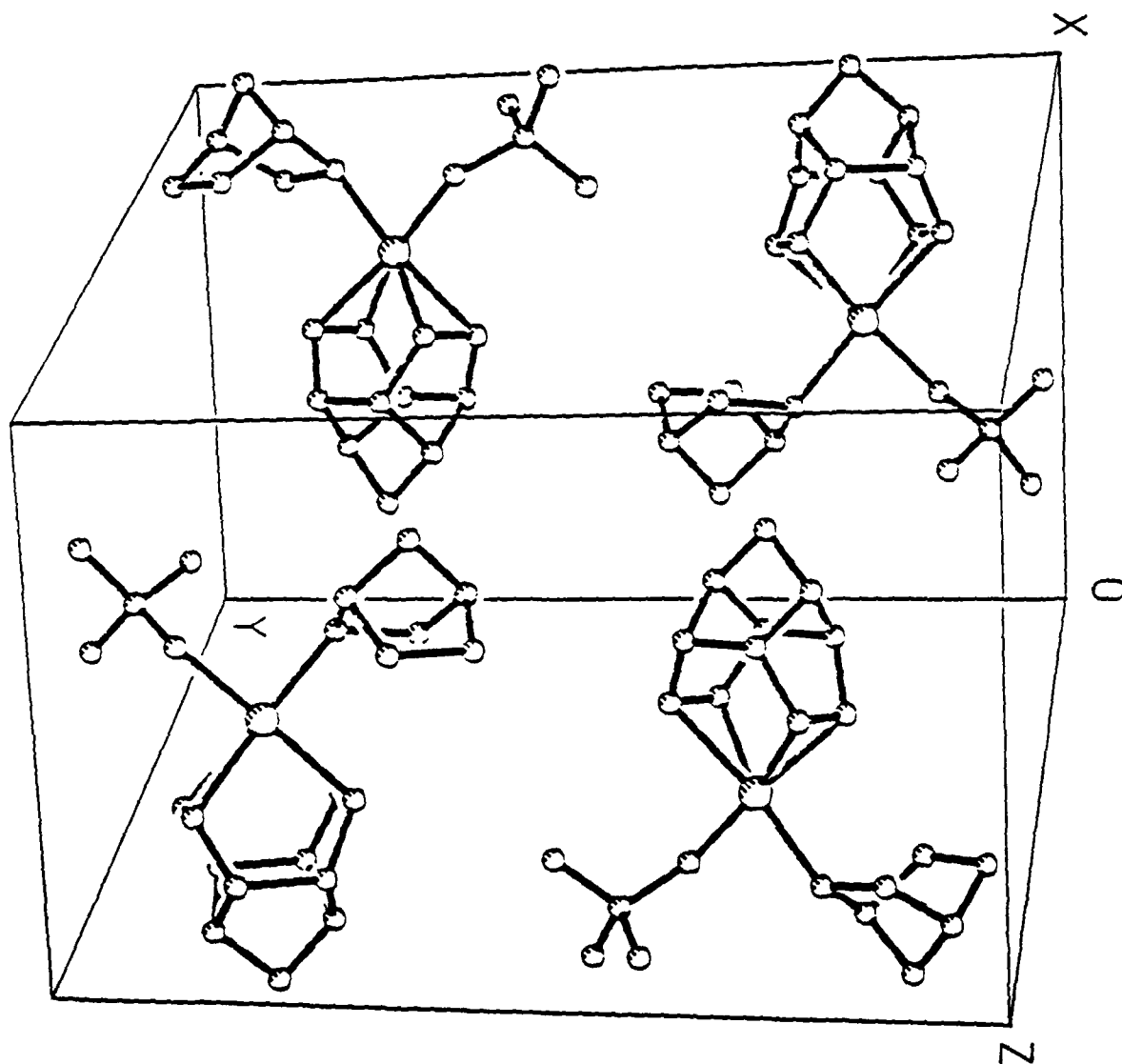| H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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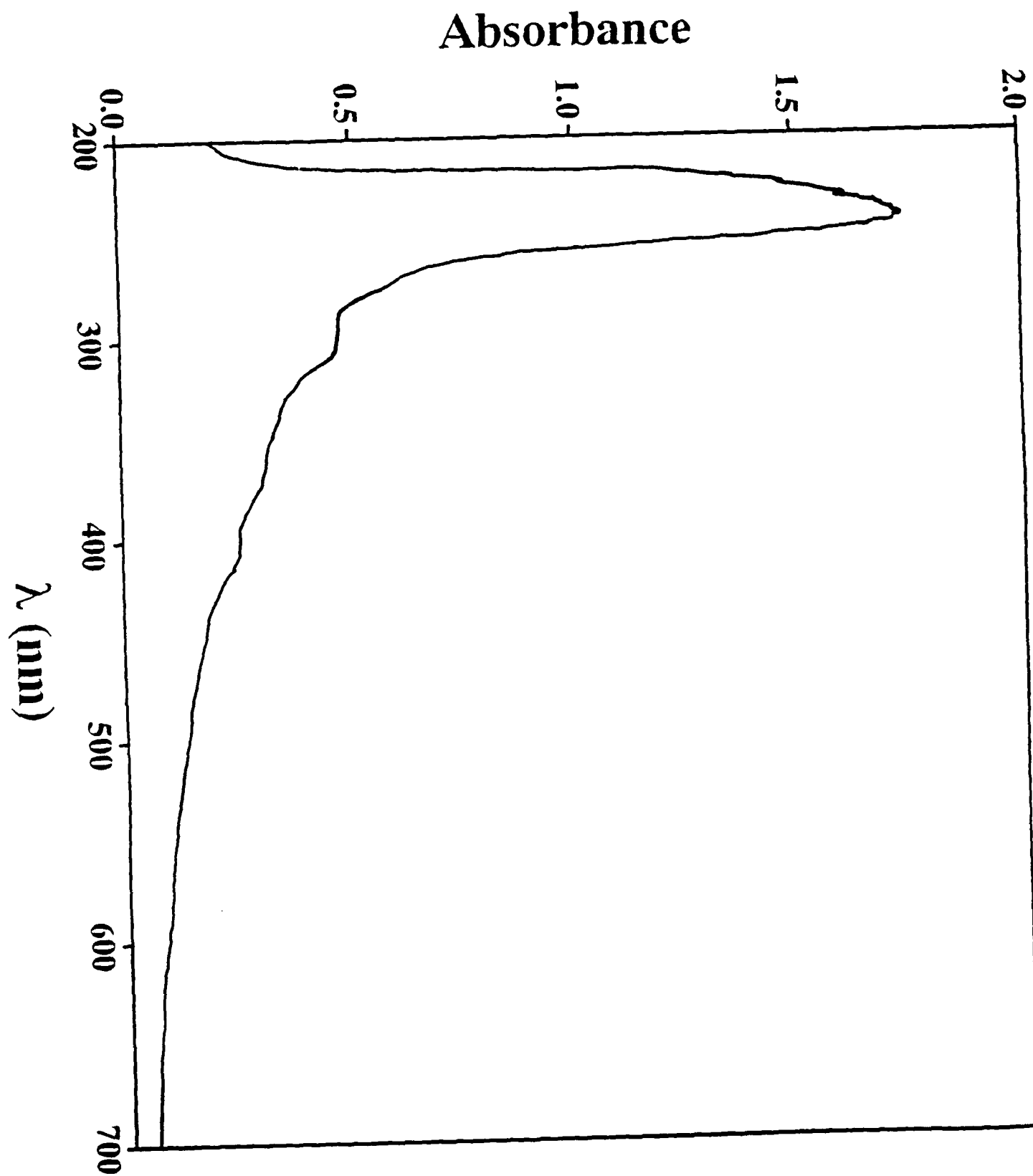
Figure 2a. Packing diagram for 2.

Figure 2b. The UV absorption spectrum of a mixture of 98% **2** and 2% **1** in *n*-hexane.

References.

(1) "P3/R3 Data Collection Manual" Nicolet Instrument Corp.: Madison, Wisconsin (1987).

(2) "SHELXTL - PLUS Users Manual" Nicolet Instrument Corp.: Madison, Wisconsin (1988).